QUALITY ASSURANCE/QUALITY CONTROL MANUAL FOR THE LOWER CAPE FEAR RIVER PROGRAM

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Version 6
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Table of Contents

Section 0 - *Version 6 changes and updates*

I. General Changes

1. Change Section Titles from center justified to left justified.
2. Review date removed from headers.
3. Section 4 was deleted and information was added to Section 1, QA/QC subcommittee list is now Appendix II. Each Section beginning with 5 changes by one number, i.e. Section 6 is now Section 5.
4. Combine Section 11 and 12, now is Section 10.

II. Section Changes

Section 1…... *Page 1* - Add information paragraph from Page 1, Section 4 which was deleted

Section 2…... No Changes

Section 3……*Page 1* - Add text description of organization
* Delete Page 2, organizational chart

Section4…… *Page 1,Paragraph 2* - change 'maintained as part of this document' to 'maintained in the UNCW-AEL'
Page 1, Paragraph 2 - change final sentence to 'Below is a list of personnel working in the UNCW-AEL at the time of this revision.'
Page 1 - Add signature sheet scan with signatures

Section 5……*Table 5.1* - Delete stations SPD, M42,BCRR,BC117, add SRWC
Figure 5.1 – Delete stations SPD, M42,BCRR,BC117, add SRWC
* Delete Figure 6.1, refer to table in Memorandum of Agreement, Appendix I

Section 6……*Page 1* - Add Safety section
Page 2 – Add protocol for measuring Secchi Depth
Pages 7-16 - Update field equipment check lists and field data sheets

Section 7……No Changes

Section 8……*Page 1* - Change Section title from 'Equipment Maintenance and Calibration" to "Multi-Parameter Water Quality Meter Operation"
Page 1 - Introduction Paragraph: Delete use of backup equipment, add procedure for malfunctioning equipment
*Delete sheets describing backup instrument use.
Page 6 - Update YSI calibration worksheet

Section 9…..*Page 1* – Update introduction paragraph
Pages 2,3,4,5 – Update Chlorophyll Grinding SOP
Page 6 – Update Chlorophyll Grinding Bench Sheet
Page 9,10 – Update Chorophyll Non-Grinding SOP

Section 10….*Page 1* – Change "principal investigators" to "Research Director"
Page 1 – Change NC DWQ to NC DWR (several places)

Table 11.1 – Delete Table, can be found in Appendix V

Section 11…. *Page 1* – Change "…technical committee members. These members will also be on the QA/QC subcommittee of the LCFRP." to "members from the QA/QC subcommittee of the LCFRP."
Page 1 – Add information regarding the use of SOP's and QA/QC manual to develop audit guidelines
*Delete all pages containing audit forms, pages 2-32

Section 12…. Update Publication List

Appendix I…… Delete actual SOPs and insert table with laboratory method codes. Move to Appendix VIII. Appendix I is now MOA.

Appendix II……Split lists into separate Appendices. Appendix II is now QAQC Subcommittee list.

Appendix III…. Move Scientist CVs to Appendix VI. Appendix III is Advisory Board List.

Appendix IV…. Benthic Ecology Lab info deleted. Appendix IV is Technical Committee list.

Appendix V……Move MOA to Appendix I. Appendix V is Subscriber list.

Appendix VI…..Move Lab Certification to Appendix VII. Appendix VI is now Scientist CVs.

Appendix VII… Lab Certification information, previously in Appendix VI.

Appendix VIII.. Lab Methods and SOPs, previously in Appendix I.

Section 1 - *Purpose of Manual*

This manual describes Quality Assurance and Quality Control (QA/QC) procedures developed for the Lower Cape Fear River Program (LCFRP) in association with the Aquatic Ecology Laboratory (AEL) at the University of North Carolina at Wilmington's Center for Marine Science (UNCW-CMS). The primary goal of this quality assurance/quality control plan is to provide for the collection, analysis and reporting of consistent and valid scientific data. These procedures are reflective of the requirements of the Memorandum of Agreement (Appendix I), the Monitoring Coalition Program Field Monitoring Guidance document from the North Carolina Division of Water Resources Coalition Monitoring Program (http://portal.ncdenr.org/web/wq/ess/eco/coalition) and the recommendations of the LCFRP QA/QC Subcommittee (Appendix II).

Periodic review is performed by the Quality Assurance/Quality Control Subcommittee and any changes are forwarded to the LCFRP Technical Committee (Appendix IV) for approval. If, at any time other than the periodic review, the Research Director determines changes are necessary, he will forward the changes to the Technical Committee. If necessary, the changes are sent to the North Carolina Department of Environment and Natural Resources, Division of Water Resources (NCDWR) coalition coordinator for review.

This QA/QC manual, Annual Reports, associated scientific publications, and relevant data are distributed to interested members of the LCFRP Advisory Board (Appendix III) and Technical Committee of the Lower Cape Fear River Program as well as other agencies and the public.

Section 2 - *Program Goals, History and Mission Statement*

1. *Program Goals*

The Lower Cape Fear River Program (LCFRP) is an un-incorporated, non-profit association. The goal of the LCFRP is to develop a science based understanding of processes that control and influence the ecology of the Cape Fear River and to provide a mechanism for information exchange and public education. In an attempt to meet this goal, a basin-wide coordinated physical, chemical, biological and water quality monitoring program has been developed and implemented. The Dischargers Association (i.e. Subscribers) contract with the Aquatic Ecology Laboratory at UNCW to carry out monitoring and research activities as specified in the Memorandum of Agreement (Appendix I) between the dischargers and the North Carolina Division of Water Resources. Monitoring sites and parameters are strategically located and established such that in-stream monitoring is more efficient, effective, basin-oriented, and yields more usable data than in-stream monitoring from individual dischargers.

2. *Program History*

In 1989/90, the Environmental Management Commission announced its intention to designate the Lower Cape Fear River as High Quality Waters. The basis of this designation was the presence of Primary Nursery Areas in the Cape Fear River Estuary. Local industries, municipalities and economic development interests were very concerned that such action would adversely impact future economic development in the area. A delay in classification was allowed based on the offer by concerned parties to obtain data and provide alternate management strategies. The local Committee of 100 took the lead and formed the Lower Cape Fear River Water Quality Study Committee which included representatives from industry, local government, environmental management, conservation, economic development and the University.

The Lower Cape Fear Committee hired a consulting firm to gather existing data and to produce a review and analysis of the current status of the river. Based on the report, the Lower Cape Fear Committee developed short and long term management plans which were submitted to the NCDWQ for review by the Environmental Management Commission. The long term plan included establishment of the Cape Fear River Program to be funded by the users and the general public. To provide objectivity, the program was to be operated by UNCW.

In the spring of 1993, the NCDWQ rejected the plan, but encouraged the establishment of the Cape Fear River Program. A group representing industry, local and state government and Chancellor Leutze from UNCW proposed an alternate plan. This plan proposed that the Cape Fear River Program be implemented as an alternative to the High Quality Waters Classification. The Environmental Management Commission accepted this plan and delayed any classification decisions.

Monitoring programs in the upper (began in January 2000) and middle (began in 1999) basins of the Cape Fear River have also been established. In order to distinguish the effort in the lower basin, the name of the program was changed to the Lower Cape Fear River Program in 1998.

3. *Mission Statement*

The mission of the Lower Cape Fear River Program is to develop an understanding of processes which control and influence the Cape Fear River and to provide a mechanism for information exchange and public education.

A. Specific Goals

- Develop, implement, and manage a basin-wide coordinated physical, chemical, and biological water quality monitoring program. Point, non-point, and naturally occurring sources will be considered in developing the monitoring plan.
- Interact with regulatory agencies, academic institutions, local industries and other groups to determine additional studies and analysis needed to develop an effective and successful management plan. Initiate the studies and assist in securing funding to conduct the research.
- Develop scientific information to provide environmental education about the basin targeting point and non-point source contributors and produce reports to identify changes or trends.
- Develop, consolidate, and maintain a data base on the Cape Fear River Basin, including historical and current data, and make data available to public and private requestors including regulatory agencies.

Section 3 - *Program Organization*

Executive Director – Dr. James Merritt, UNCW Center for Marine Science

Advisory Board (Appendix III)

- Determines policy for the program.
- Consists of representatives from citizen's groups, academia, local government, industries, the business community and regulatory agencies.

Technical Committee (Appendix IV)

- Developed and oversees the monitoring program and scientific aspects of the program.
- Consists of representatives from UNCW, the NC Division of Water Resource's Monitoring Coalition Program, the NC Division of Marine Fisheries, the Cape Fear Public Utility Authority, the US Army Corps of Engineers, technical representatives from industries, New Hanover County, US Geological Survey and other interested organizations and municipalities.

QA/QC Subcommittee (Appendix II)

- A subcommittee of the Technical Committee created to develop and maintain a Quality Assurance/Quality Control program to ensure scientific accuracy and precision and compliance with the Memorandum of Agreement (Appendix I).

Research and Monitoring Program

- Research Director – Dr. Michael A. Mallin, Aquatic Ecology Lab at UNCW

- Benthic Research Coordinators –Dr. Martin Posey, Benthic Ecology Lab, UNCW
  Troy Alphin, Benthic Ecology Lab, UNCW

- Sampling Coordinator – Matthew McIver, Aquatic Ecology Lab at UNCW

Section 4 - *Scientists*

Scientific staff is hired on a consensus basis between Dr. Michael Mallin and Dr. James F. Merritt. Personnel are hired on the basis of appropriate education, skills, field and laboratory experience. Curriculum vitae for these personnel are included in Appendix VI of this document.

A list of personnel, dates of employment and each employee's signature is maintained in the AEL-UNCW. The list is used to verify signatures and initials on data sheets and other LCFRP documents. Non-program scientists in the Aquatic Ecology Laboratory routinely assist with fieldwork, analysis, and data entry. The signatures of these employees are also included. Below is a list of personnel working in the AEL-UNCW at the time of this revision.

Lower Cape Fear River Program Scientists:

Dr. Michael Mallin, Science Director, 1994-present

Matthew McIver, Sampling Coordinator/QAQC, 1994-present

Aquatic Ecology Lab Scientists:

Amanda Kahn Dickens, PhD, 2009-present

Rena H. Turner, 2004-present

Anna Robuck (Masters Student), 2011-present

Mary Grace Lemon (Masters Student), 2013-present

Section 5 - *Station Descriptions and Sampling Schedule*

Samples are collected at 32 stations required by the 2011 Memorandum of Agreement with North Carolina Division of Water Resources (Appendix I, Table A-1 Revised) and one additional research station added by the AEL (Table 5.1 and Figure 5.1). Samples are collected monthly requiring five field days. There are nine estuarine stations sampled by boat, five riverine stations sampled by boat and nineteen stations sampled from highway bridges and culverts. Additional sampling is carried out during summer as required by the Memorandum of Agreement.

Table 5.1 Station information for the Lower Cape Fear River Program.

Collected by Boat

AEL Station	DWR Station #	Description	Comments	County	Lat	Lon	Stream Class.	HUC
NC11	B8360000	Cape Fear River at NC 11 nr East Arcadia	Below Lock and Dam 1, Represents water entering lower basin	Bladen	34.3969	-78.2675	WS-IV Sw	03030005
LVC2	B8441000	Livingston Creek at Momentive Walkway nr Acme	DWR ambient station, Downstream of Momentive	Columbus	34.3353	-78.2011	C Sw	03030005
AC	B8450000	Cape Fear River at Neils Eddy Landing nr Acme	1 mile below IP, DWR ambient station	Columbus	34.3555	-78.1794	C Sw	03030005
DP	B8465000	Cape Fear River at Intake nr Hooper Hill	AT DAK intake, just above confluence with Black R.	Brunswick	34.3358	-78.0534	C Sw	03030005
BBT		Black River below Lyons Thorofare	UNCW AEL station	Pender	34.3513	-78.0490	C Sw ORW+	0303005
IC	B9030000	Cape Fear River ups Indian Creek nr Phoenix	Downstream of several point source discharges	Brunswick	34.3021	-78.0137	C Sw	0303005
NAV	B9050025	Cape Fear River dns of RR bridge at Navassa	Downstream of several point source discharges	Brunswick	34.2594	-77.9877	SC	0303005
HB	B9050100	Cape Fear River at S. end of Horseshoe Bend nr Wilmington	Upstream of confluence with NE Cape Fear River	Brunswick	34.2437	-77.9698	SC	0303005
BRR	B9790000	Brunswick River dns NC 17 at park nr Belville	Near Belville discharge	Brunswick	34.2214	-77.9787	SC	03030005
M61	B9800000	Cape Fear River at Channel Marker 61 at Wilmington	Downstream of several point source discharges	New Hanover	34.1938	-77.9573	SC	03030005
M54	B9795000	Cape Fear River at Channel Marker 54	Downstream of several point source discharges	New Hanover	34.1393	-77.946	SC	03030005
M35	B9850100	Cape Fear River at Channel Marker 35	Upstream of Carolina Beach discharge	Brunswick	34.0335	-77.937	SC	03030005
M23	B9910000	Cape Fear River at Channel Marker 23	Downstream of Carolina Beach discharge	Brunswick	33.9456	-77.9696	SA HQW	03030005
M18	B9921000	Cape Fear River at Channel Marker 18	Near mouth of Cape Fear River	Brunswick	33.913	-78.017	SC	03030005
NCF6	B9670000	NE Cape Fear nr Wrightsboro	Downstream of several point source discharges	New Hanover	34.3171	-77.9538	C Sw	0303007

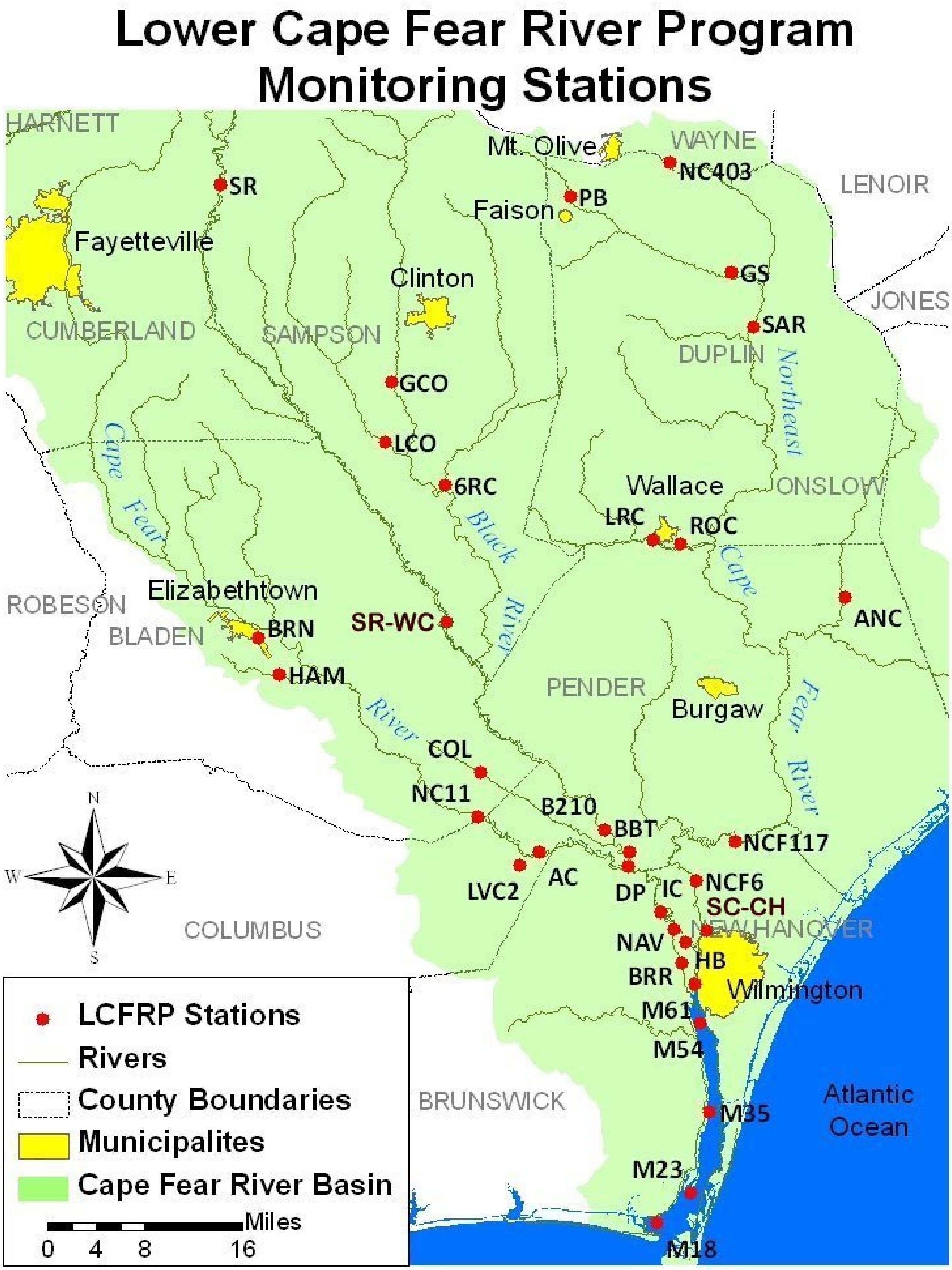
Collected by Land

6RC	B8740000	Six Runs Creek at SR 1003 nr Ingold	Upstream of Black River, CAFOs in watershed	Sampson	34.7933	-78.3113	C Sw ORW+	03030006
LCO	B8610001	Little Coharie Creek at SR 1207 nr Ingold	Upstream of Great Coharie, CAFOs in watershed	Sampson	34.8347	-78.3709	C Sw	03030006
GCO	B8604000	Great Coharie Creek at SR 1214 nr Butler Crossroads	Downstream of Clinton, CAFOs in watershed	Sampson	34.9186	-78.3887	C Sw	03030006
SR	B8470000	South River at US 13 nr Cooper	Downstream of Dunn	Sampson	35.156	-78.6401	C Sw	03030006
BRN	B8340050	Browns Creek at NC87 nr Elizabethtown	CAFOs in watershed	Bladen	34.6136	-78.5848	C	03030005
HAM	B8340200	Hammond Creek at SR 1704 nr Mt. Olive	CAFOs in watershed	Bladen	34.5685	-78.5515	C	03030005

Collected by Land

AEL Station	DWR Station #	Description	Comments	County	Lat	Lon	Stream Class.	HUC
COL	B8981000	Colly Creek at NC 53 at Colly	Pristine area	Bladen	34.4641	-78.2569	C Sw	03030006
B210	B9000000	Black River at NC 210 at Still Bluff	1st bridge upstream of Cape Fear River	Pender	34.4312	-78.1441	C Sw ORW+	03030006
NC403	B9090000	NE Cape Fear River at NC 403 nr Williams	Downstream of Mt. Olive Pickle, CAFOs in watershed	Duplin	35.1784	-77.9807	C Sw	0303007
PB	B9130000	Panther Branch (Creek) nr Faison	Downstream of Bay Valley Foods	Duplin	35.1345	-78.1363	C Sw	0303007
GS	B9191000	Goshen Swamp at NC 11 and NC 903 nr Kornegay	CAFOs in watershed	Duplin	35.0281	-77.8516	C Sw	0303007
SAR	B9191500	NE Cape Fear River SR 1700 nr Sarecta	Downstream of several point source discharges	Duplin	34.9801	-77.8622	C Sw	0303007
ROC	B9430000	Rockfish Creek at US 117 nr Wallace	Upstream of Wallace discharge	Duplin	34.7168	-77.9795	C Sw	0303007
LRC	B9460000	Little Rockfish Creek at NC 11 nr Wallace	DWR Benthic station	Duplin	34.7224	-77.9814	C Sw	0303007
ANC	B9490000	Angola Creek at NC 53 nr Maple Hill	DWR Benthic station	Pender	34.6562	-77.7351	C Sw	0303007
SR WC	B8920000	South River at SR 1007 (Wildcat/Ennis Bridge Road)	Upstream of Black River	Sampson	34.6402	-78.3116	C Sw ORW+	03030006
NCF117	B9580000	NE Cape Fear River at US 117 at Castle Hayne	DWR ambient station, Downstream of point source discharges	New Hanover	34.3637	-77.8965	B Sw	0303007
SC-CH	B9720000	Smith Creek at US 117 and NC 133 at Wilmington	Urban runoff, Downstream of Wilmington Northside WWTP	New Hanover	34.2586	-77.9391	C Sw	0303007

Figure 5.1 LCFRP Station Map



Section 6 - *Standard Operating Procedures for Field Sampling*

Water samples are collected and delivered to a state certified laboratory to be analyzed for fecal coliform/enterococcus bacteria, heavy metals, total suspended solids, lab turbidity and nutrients. MOA required chlorophyll *a* samples are analyzed in the AEL using NC DWR certified procedures. Non-MOA required chlorophyll *a* and Biochemical Oxygen Demand samples are analyzed in the AEL-UNCW using non-certified procedures. In situ readings of temperature, pH, conductivity, dissolved oxygen, turbidity and salinity are measured using a YSI Multi Parameter Water Quality Meter (MPWQM) at the same location as the bottle samples are collected. The AEL-UNCW is NC DWR certified for field parameters and chlorophyll *a* analysis (Appendix VI). Standard Operating Procedures (SOPs) are reviewed and revised periodically as needed and the revision dates are documented in the preface to the SOPs.

1. Safety
   Safety should be a primary concern during monitoring activities. Recommended safety practices include but are not limited to:
   - Follow all traffic regulations.
   - Be aware of surrounding conditions i.e. weather, animals, plants and take appropriate precautions.
   - Park safely on the side of highways, wear safety vests and use vehicle blinkers or safety cones
   - A minimum of two people should be present on boat sampling excursions and all personnel should wear personal floatation devices.
   - If someone feels that a station is unsafe, the coalition coordinator should be notified to discuss possible alternate sampling ideas.
   - Never wade into high water or swim to collect samples.

2. Quality Assurance During Sample Collection and Transport
   The purpose of collecting samples is to obtain a representative portion of the medium being evaluated. Proper sampling procedures and the handling of samples after collection cannot be overemphasized. Required Chain of Custody forms and procedures should be employed (Section 7).

3. General Sample Collection Methodology
   - Refer to the appropriate field equipment checklist prior to each sampling trip.
   - The preferred method of collecting a sample is to collect the water directly in the stream, in the container used to transport the sample to the laboratory.
   - During in-stream sampling hold bottle at base and submerge into water neck down, quickly turn the bottle until the mouth of the bottle is tilted up and facing into the current, approximately 0.15 m below the surface. If no current is present move the bottle forward in the upstream direction. When using bottles that are pre-loaded with preservative, i.e. nutrient bottles with H_2SO4, submerge the neck up bottle under the water quickly. If sampling from a boat fill the bottles as to avoid collecting any compounds coming from the boat.
   - In instances when a bucket must be used, rinse the bucket thoroughly with water from the site prior to sample collection making sure to dump rinse water away from sampling area.
   - When sampling from a bridge collect samples midstream on upstream side of the bridge.
   - If using the Remote Bottle-Filling Device (RBFD), Coli-sampler or Long-arm (Figures 6.1, 6.2, 6.3) lower device to the surface and rapidly submerge so bottles fill below the surface.
   - Be sure to leave ample air space in bottles to facilitate mixing by shaking.

- Place samples in coolers, on ice, for storage and transport to the lab.

4. Parameter Specific Sampling Procedures

A. Chlorophyll *a*

MOA required samples at stations M18, M61, NC11 - DWR sampling protocol requires photic zone integrated sampling at stations where this sampling is feasible, i.e. depth greater than 1 meter, which includes all three of the LCFRP sites. Photic zone samples are collected using the lab-line sampler (Figure 6.4). The secchi depth is measured and multiplied times 2 to calculate the photic zone depth. Lower the instrument to this depth, pop the plugs and pull it slowly to the surface. Station specific brown chl *a* bottles are filled directly from the lab-line. Rinse the lab-line sampler with distilled water between stations.

Non-MOA samples - Chlorophyll *a* samples are collected in triplicate in 120 ml brown plastic bottles that are clean and dry. Each bottle is filled separately about 0.25 m below the surface.

B. Secchi Depth

Secchi Depth is collected as supplementary data, not required by the MOA. The secchi disc is mounted on an extending pole, marked in tenths of meters. Extend the pole and lower into the water on the shady side of the boat. The last depth at which you can see the white versus black markings is recorded as the Secchi Depth.

C. Biochemical Oxygen Demand

BOD samples are collected in 1 L plastic bottles that have been cleaned (24 hours in 10% HCl bath) and dried. Analysis must begin within 24 hours of collection.

C. Fecal Coliform Bacteria/Enterococcus

Fecal coliform samples are collected in sterilized plastic 120 mL bottles from the contract lab. Sample must be taken directly in stream using Coli-sampler when unable to reach water. Analysis must begin within 8 hrs of collection.

D. Metals- Metals Sampling has been suspended by NC DWR till further notice

Metals samples are collected in a liter plastic bottle containing nitric acid. Samples are stored on ice during transport to the laboratory. Mercury samples may be held for no more than 28 days. Samples analyzed for metals other than mercury may be held for no more than six months from date of collection. Samples must be taken directly in stream using the RBFD when necessary.

E. Total Suspended Solids

Aliquots of water for total suspended solids measurements are removed from non-preserved sample bottles provided by the contract lab. Every attempt is made to analyze samples within 24 hours, but in no case are samples held more than 7 days.

F. Nutrients-Nitrate/Nitrite, Total Kjeldahl Nitrogen, Ammonium, Total Phos.

All nutrients are analyzed from the same sample, collected in plastic liter bottles containing sufficient H_2SO_4 to preserve sample at a pH from 1.5 to 2 (*Bottles are provided by and acidified by the contract lab). Nitrate samples will be processed within 48 hours of sampling. TKN, ammonium and total phosphorus samples will be processed within 28 days of collection. **Nutrients at stations with required chl *a* sampling should be collected as photic zone integrated samples using the lab-line. See section A. for information on using the lab line.

G. Turbidity

Laboratory turbidity samples are collected in 8 oz. plastic bottles, hold time is 48 hours. Field turbidity (not a state certified parameter) is measured with the YSI multi-parameter water quality meter. Prior to leaving for the field the meter is calibrated at 0 NTU. Upon return to the laboratory the meter is checked using the 0 NTU standard.

5. Field Parameters
   - Water temperature*, dissolved oxygen*, specific conductivity*/salinity, field turbidity and pH* (*NC DWR certified parameters) are measured using a YSI multi-parameter water quality meter (MPWQM).
   - Measurements are taken near the surface at approximately 0.15 m and near the bottom of the water column at N11, AC, DP, BBT, IC, NCF6, NAV, HB, BRR, M61, M54, M35, M23 and M18 and at the surface for all other stations.
   - Data are recorded on pre-printed field data sheets.
   - Probes are calibrated according to manufacturer’s instructions and DWR certification guidelines before each sampling trip and are checked upon return to the lab (see Section 8 for calibration information). Calibration worksheets are completed and attached to the field data sheet for each sampling trip.
   - A dissolved oxygen calibration drift check is performed during each trip as indicated on the field data sheet. Put the cap on the sonde, allow to equilibrate for 15 minutes and record % saturation value.
   - In the event the MPWQM fails in the field scientists must return to the lab to get a new instrument or plan to resample on another day.

Figure 6.1 Long-arm Sampling Pole

This device holds bottles of all sizes and is used to extend out so that a bottle may be filled directly in-stream when access to the water body is difficult.

<u>Figure 6.2 Remote Bottle Filling Device (RBFD)</u>
This device is constructed of plastic, rope, and a rubber coated weight. Its primary purpose is for in-stream water sampling when access to the water is not possible, i.e. from a bridge. The bottles are placed on the platform and secured with the bottle hood, caps extruding from the upper holes. The hood bungees secure the hood. The apparatus is lowered to the water surface and rapidly lowered so that the bottles fill below the surface.

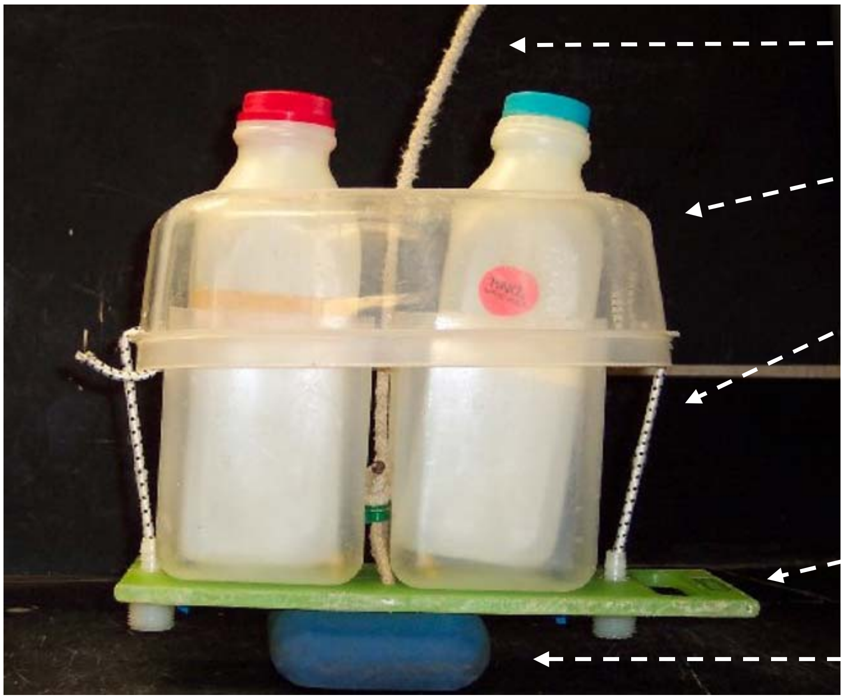


Figure 6.3 Remote Coliform Sampler
This device is constructed of pvc, rop, and a weight. Its primary purpose is for in-stream water sampling when access to the water is not possible, i.e. from a bridge, and when the bottles do not fit in the RBFD (Figure 7.2). The bottles are placed in the top and secured with a rubber band. The apparatus is lowered to the water surface and rapidly lowered so that the bottles fill below the surface.

Figure 6.4 Lab-Line Integrated Sampler
This device is constructed of plastic, rope, and a spring. Its purpose is to collect an integrated sample of water over a given depth. The bottle plugs are put into the holes on the top, the instrument is lowered to twice the secchi depth, a quick pull on the line pops the plugs out and as the instrument is raised to the surface, it fills with water over the entire depth range.

Typical Field Equipment Checklist:

Field Equipment Checklist for Lower Cape Fear River Program Sampling

Estuary Boat Trip

NAV HB BRR M61 M54 M35 M23 M18

(1) Quart Bottle Unpreserved per station (TSS)
(1) Quart Bottle w/H_2SO_4 per station (Nutrients)

Label M61 and M18 with green plastic tie to remind for Lab Line use

(1) Pint bottle per station for NAV, M61, M18 (Lab Turbidity)
(1) Coliform(NAV HB)/Enterococcus (M18 M23 M35 M54 M61 BRR)
(3) 125 ml Chlorophyll *a* Bottles per station
(1) 250 ml Chl *a* bottle for M18
125 ml Chl *a* bottle for M61

Sample with Lab Line, Label with green tie

___ Float Plan
___ YSI MPWQ Probe
___ YSI Guard
___ YSI Cap
___ Lab-line sampler
___ Light Meter
___ Field Notebook and Pens
___ Field Data Sheets
___ Chain of Custody Sheets
___ Camera
___ Field Gloves
___ Cellular Phone
___ Check engine position
___ Binoculars
___ FATES bag
___ Temperature Bottle
___ Rain Gear/Float Coats
___ Red Bucket
___ Small Bucket
___ YSI Line – **15m**
___ 2 Weights: YSI and Light Meter
___ 3 Large Coolers
___ Ice the Coolers
___ Secchi Disk (On Boat)
___ Fill-out Field Info Board
___ Post Float Plan at Boat Shop
___ Hand towel
___ Check for Boat Box and Keys
___ Call EChem 392-0223
___ Boat Gas Card
___ Chla chain of custody
___ Di water to rinse LabLine

Typical Field Data Sheet:

LOWER CAPE FEAR RIVER PROGRAM - Field Data									
Date -				Scientists-					
Weather/Tide-				Recorder Signature-					
Station	Depth meters	Temp °celcius	pH	Spec Cond µS	Salinity psu	Turbidity NTU	Diss Ox mg/L	DO Satur %	Light Att.
M18	0.1								
- time									
- secchi									
-bottom									
M23	0.1								
- time									
- secchi									
-bottom									
M35	0.1								
- time									
- secchi									
-bottom									
M54	0.1								
- time									
- secchi									
-bottom									
Dissolved Oxygen drift check (95-105%) ______________									
BRR	0.1								
- time									
- secchi									
-bottom									
M61	0.1								
- time									
- secchi									
-bottom									
HB	0.1								
- time									
- secchi									
-bottom									
NAV	0.1								
- time									
- secchi									
-bottom									

Section 7 - *Chain of Custody*

Chain of custody procedures are utilized in order to document the handling of samples from the time of collection through processing in the laboratory. This process provides written documentation that can be used to trace the progress of the sample for quality assurance purposes. Chain of Custody forms are customized for each trip for both Contract Lab COCs and AEL COCs (see pages 2 and 3 for examples).

Sample Custody Procedures

1. Bottles are labeled with station name, date, preservative type and analysis parameter in the lab prior to the sampling trip.

2. Bottles are filled at the water body at the previously selected stations. Chain of Custody sheets will be completed reflecting the types and number of samples collected at each station as well as date, time, initial temperature and preservative. COC sheets are pre-populated and can be printed in the lab prior to each trip.

3. Field investigators will keep the samples in their custody until they are delivered to the processing laboratory or relinquished at specified meeting point.

4. A chain of custody form shall accompany all samples. When possession of samples is transferred, the individual receiving and the individual relinquishing/delivering the samples will sign and date the form.

envirochem

ENVIRONMENTAL CHEMISTS, INC

Sample Collection and Chain of Custody

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Wilmington, NC 28405
Phone: (910) 392-0223
Fax (910) 392.4424
EMAIL: ECHEMW@aol.com

1

Client: UNCW – CMS Lower Cape Fear River Program – Estuary Boat Trip
Collected By: **Report No:**________________
Sample Type: I = Influent, E = Effluent, W =Well, ST =Stream, SO =Soil, SL= Sludge Other:______________________

Sample Identification	LAB ID NUMBER	Collection			Sample Type	Composite or Grab	Container (P or G)	Chlorine mg/L	PRESERVATION							ANALYSIS REQUESTED
		DATE	TIME	TEMP					NONE	HCL	H_2SO_4	HNO_3	NaOH	THIO	OTHER	
NAV						G	P		X							TSS/Turbidity
						G	P				X					NO_2+NO_3, TKN, Total P, Ammonia
						G	P							X		Fecal
						G	P									
HB						G	P		X							TSS
						G	P				X					NO_2+NO_3, TKN, Total P, Ammonia
						G	P							X		Fecal
						G	P									
BRR						G	P		X							TSS
						G	P				X					NO_2+NO_3, TKN, Total P, Ammonia
						G	P							X		Fecal
						G	P									

NOTICE – DECHLORINATION : Samples for Ammonia, TKN, Cyanide, Phenol, and Bacteria must be dechlorinated (0.2 ppm or less) in the field at the time of collection. See reverse side for instructions.

Transfer	Relinquished By:	Date/Time	Received By:	Date/Time
1.				

Temperature when Received:__________ **Accepted:**___________ **Rejected:**__________ **Resample Requested:**____________________
Delivered By:_________________________ **Received By:**__________________________ **Date:**__________**Time:**_________________
Comments: ____

COC 1.2.1 Chlorophyll *a* Chain of Custody for Grinding Analysis Samples **-LCFRP**

Instructions: Initials, Date and Time are required for each action.

Month/Year		
	M18	**M61**
Collected By: Date/Time		
Received on Ice		
Duplicates? Yes or No		
Filtered By:		
Filters in Freezer time		

Section 8- *Multi-Parameter Water Quality Meter Operation*

A Multi Parameter Water Quality Meter (MPWQM) consists of a hand held display unit, a cable and a sonde equipped with parameter specific probes. Each probe is calibrated before sampling activities and checked upon return to the laboratory according to procedures outlined by the NC DWR Laboratory Certification program and the Monitoring Coalition Program Field Monitoring Guidance document from the North Carolina Division of Water Resources Coalition Monitoring Program (http://portal.ncdenr.org/web/wq/ess/eco/coalition). Calibration data is recorded on a calibration worksheet and kept on file in the laboratory attached to the field data sheets. Backup meters, carried in the field in previous years, are no longer used. These instruments, due to their rare use, were usually un-reliable. If the MPWQM malfunctions in the field the protocol is to return to collect data on another day.

Aquatic Ecology Laboratory

SOP 1.1 YSI Multi-parameter Water Quality Sonde Operation for NC DWQ Field Certification Compliance

Development date – October 2007	Author(s) – *Scott Ensign, Matthew McIver*
Revised – October 2014, Revision 4	Author(s) – *Matthew McIver*
Reference(s) – YSI 6820/6920 Sonde/650 MDS operation manuals NC DWR Lab Certification Webpage http://portal.ncdenr.org/web/wq/lab/cert	

I. Introduction

Purpose: The quality and accuracy of data obtained using a multi-parameter water quality sonde is dictated by several factors including the proper functioning of the individual probes, the calibration procedure and the use of the instrument when taking measurements. This protocol is designed to comply with the rules of the North Carolina Division of Water Resource's field/lab certification program and must be performed each day measurements are made. Using this protocol ensures not only quality and accurate data, but also documentation of the procedures used to obtain this data. This protocol is specifically indicated for the use of the YSI 6820/6920 sonde with a 650 MDS display.

II. Start Up

The YSI 650 display unit batteries automatically discharge over time, therefore charging for 12-24 hours prior to sampling is recommended. The unit should not be left charging for periods of more than 24 hrs. Be sure to power off the YSI 650 before leaving it to charge.

1. Print out WS 1.1.1 YSI Calibration Worksheet to record calibration metrics. This sheet is to be stapled to the field data sheet for each day of sampling.
2. Remove the sonde cap and inspect each probe. Check the dissolved oxygen probe membrane for tears, wrinkles or bubbles (any of these will require membrane replacement). DAB the membrane with a lab tissue to remove moisture.

3. Replace the cap leaving it loosely attached during calibration so that any barometric pressure changes can equilibrate inside the cap. Make sure the sponge is moist.
4. Connect the cable to the sonde and power up the YSI 650. The unit will display the *650 Main Menu*.
5. A dissolved oxygen probe reverse warm-up check should be performed to ensure that the DO probe is functioning properly: highlight *Sonde run*, press ← and watch the DO saturation value. The value should start high and decrease towards 100%. If the number increases in value there may be a problem with the DO probe and the DO charge and gain need to be monitored when calibrating.
6. Press *Esc* to exit to *650 Main Menu*.

III. Calibration

Highlight *Sonde Menu* and press ← (enter button) to enter *Main (sonde)* menu. Enter *Report* and turn on the DO charge and pH mV functions. Escape to Main (sonde) and enter *Calibrate menu.*

A. Dissolved Oxygen Calibration
   1. Dissolved Oxygen is calibrated using air saturation inside the sonde cap. DAB the probe membrane to remove moisture and loosely screw on cap with on loosely with a wet sponge inside.
   2. Enter the *Dissolved Oxy menu*, then enter *DOsat %*. Barometric pressure is entered in mmHg (mmHg = inHg x 25.4) and recorded on the calibration worksheet. Press ← to begin calibration.
   3. Observe DO% parameter display for about 15 minutes and when this value has stabilized record this pre-cal value and the temperature value on the calibration worksheet, then press ←. If the calibration was successful *Calibrated* will appear at the top of the screen. Record the cal % saturation value and the DO charge on the calibration worksheet. The DO charge should be between -50 and 50, if not the probe may be bad, consult the maual. Press Esc thrice to return to the *Main* menu, highlight *calibrate* and press ← to enter calibration menu.

B. pH Calibration

   If expected field pH values will fall between either 4 – 7 or 7-10, then you can perform a 2 point calibration with the two buffers that bracket your values. In the case of potential field values spanning 4-10 you should perform a three point calibration using 7 buffer first, then 4 and 10.
   1. To begin the pH calibration select the *ISE1 pH* option from the *Calibrate* menu then select the *2 Point or 3 point* option. The display will prompt you to enter the first pH.
   2. Put pH 7 buffer in the calibration cup (medicine dosage cup) and slip over the pH probe and the temperature thermistor making sure both are submerged.
   3. Press ← and observe the pH parameter display. When the pH value has stabilized record the pre-cal value and the temperature on the calibration worksheet, then press ←. If the calibration is successful *Calibrated* will appear at the top of the screen. Record the cal value and the millivolt value on the calibration worksheet. Millivolt values outside of the acceptable ranges may indicate a problem with the probe.
      pH millivolt acceptable ranges: *buffer 7 → 0 +/- 50*
      buffer 4 →180 +/- 50
      buffer 10 → -180 +/-50

   4. Press ← and the display will prompt you to enter a second pH standard value. Remove the calibration cup, rinse the probes with DI water, dry the probes with a lab tissue and place the calibration cup with the second pH buffer solution onto the probes.

5. Press ← and when the pH value has stabilized record the pre-cal value and the temperature value then press ←. If the calibration is successful *Calibrated* will appear at the top of the screen. Record the cal value and the pH millivolt value and check acceptable ranges.
6. Repeat process for third pH buffer is necessary, otherwise press Esc twice to return to the Calibrate menu so that you can begin the next calibration.

C. Specific Conductivity Calibration

Calibration solutions should be made with KCl (potassium chloride). A one point calibration is performed using a 1,430 micro-siemens standard. For specific situations where the measured value will be within a small range you may select a standard that more closely matches that range.

1. From the *Calibrate* menu choose *Conductivity*, from the *Conductivity menu* choose *SpCond*.
2. Enter the standard value in milli-Siemens/cm.
3. Fill the conductivity calibration tube with 12 mls of used calibration solution and slide the tube onto the Conductivity probe to rinse.
4. Dump the used solution, refill with fresh solution and insert onto probe. Ensure that the round hole on the conductivity probe is covered with calibration solution.
5. Press → and watch the display for temperature and conductivity values to equilibrate. When the reading is stable record the pre-cal value and temperature, then press →.
6. If the calibration is successful *Calibrated* will appear at the top of the screen. Record the cal value.
7. Press Esc thrice to return to the *Main* menu and scroll down to highlight the *advanced* menu, press → and select cal constants.
8. Record the conductivity cal constant which should be 4.5 to 5.5, if not the probe may be bad.
9. Esc once, select *report* and turn off the DO charge and pH millivolt readings.
10. Remove the calibration tube, pour used solution into the used container and rinse both the tube and the probe with distilled water. Press Esc twice to return to the *calibrate* menu.

D. Turbidity Calibration

A one-point calibration is performed using a 0 NTU standard (distilled water).

1. Fill the bottom portion of the calibration cup with distilled water. Screw the calibration cup to the YSI sonde body.
2. From the *Calibrate* menu choose *Turbidity*, then choose *1 point*. Enter 0 for the standard value and press →.
3. Observe the turbidity value and select *clean optics* at the top of the screen to wipe the probe.
4. When the value stabilizes record the pre-cal value, then press →. If the calibration is successful *Calibrated* will appear at the top of the screen. Record the cal value.
5. Press Esc twice to return to the Calibrate menu. Remove the calibration cup and discard solution.

IV. Post Sampling Calibration Check

Lab certification rules dictate that after data collection the multi-parameter water quality sonde should be checked to make sure the calibration has not drifted out of acceptable ranges.

A. Dissolved Oxygen

The dissolved oxygen calibration is checked at 100% air saturation. Dab the membrane with a lab tissue and screw storage cap on loosely. After a 15 minute equilibration period record the appropriate data on the worksheet. The DO percent saturation acceptance range is 95% to 105%.

B. pH
The pH calibration should be checked with two buffers that bracket the values read in the field. The acceptable range is within 0.1 standard units.

C. Specific Conductivity
The specific conductivity calibration is checked with two buffers which bracket the values read in the field. The acceptable range is within 5% of the standard value.

D. Turbidity
Although there is no certification for turbidity, a check using 0 NTU standard is performed.

V. Calibration Standards

MPWQS calibration is only good if the calibration standards are good. The AEL orders most of its calibration standards from EXAXOL Chemical Corporation (14325 60th St. N., Clearwater, FL 33760, 727-524-7732). Expiration dates should be noted and highlighted. A calibration standards use log (LS 1.9.1) is maintained and should be filled out as standards are opened and discarded.

VI. YSI Cleaning, Maintenance and Storage

A. Cleaning
The YSI sonde probes may be cleaned periodically using SOP 3.5. Record date of cleaning on the YSI cleaning log LS 3.5.1

B. Maintenance
Maintenance procedures for each probe are outlined in the YSI user's manual. The dissolved oxygen membrane should be changed on a regular basis, otherwise most probes do not need regular maintenance.

C. Storage
YSI sondes are stored by hanging from the stainless steel hooks under the shelving. The storage cup containing a damp sponge is screwed on snug. When a sonde will not be used for more than several weeks long term storage procedures are used:

pH- Remove the pH probe, store in the storage vial containing . Plug pH port.
Turbidity- Remove the turbidity probe and store dry. Plug turbidity port.
Dissolved Oxygen- Leave the probe on the sonde. Put distilled water in the storage cup so that the end of the do probe is in the water. This keeps the probe end from drying out during storage which may cause small cracks to form.
Conductivity- Leave the conductivity probe on the sonde during long term storage.

WS 1.1.1 YSI Calibration Worksheet

Project-

Date-

Analyst Signature-

Sonde Number-

Dissolved Oxygen

	AEL Probe # -			Startup test- pass fail		DO charge (50 +/- 25) =
	Time	Barometric Pressure (mm Hg)	Air Temperature (°C)	Initial % Saturation	After Cal. % Saturation	DO in mg/L
Calibration						
Post Sampling Check						
				within 95-105%? - yes no		

pH

	AEL Probe # -					
	Time		Temperature (°C)	Initial Reading	After Cal. Reading	Millivolt Reading
Calibration		Buffer 7.0				
		Buffer 4.0				
		Buffer 10.0				

	Time		Temperature (°C)	Reading	Within 0.2 units ?
Post-Sampling Check		Buffer ___			yes no
		Buffer ___			yes no

Specific Conductivity

	AEL Probe # -			Cell Constant (5.0 +/- 0.5)=		
	Time	Standard Value µS	Temperature (°C)	Initial Reading	After Cal. Reading	
Calibration		14				
Check Standard					within 10% yes no	
Post Sampling Check					within 10% yes no	
					within 10% yes no	

Turbidity

	AEL Probe # -			
Calibration	Time	Standard Value NTU	Initial Reading	After Cal. Reading
Post Sampling Check				within 3 to -3 NTU? yes no

Section 9 - *Standard Operating Procedures for AEL Lab Analyses*

Several parameters are analyzed by the Aquatic Ecology Laboratory at the UNCW Center for Marine Science. The MOA requires Chlorophyll *a* analysis using NC DWR Certified methodology (employs grinding of the filter) at three LCFRP stations. The Aquatic Ecology Laboratory is NC DWR certified to perform this analysis (Appendix VII). Chlorophyll *a* is also analyzed for all LCFRP stations using a non-grinding method. Biochemical Oxygen Demand (5 and 20 day), which is not required by the MOA, is analyzed at six LCFRP stations using non-certified methods. All other Memorandum of Agreement required analyses are performed by a state certified analytical laboratory. SOPs and laboratory data sheets for the AEL performed analyses are provided here. Contract laboratory methods are included as Appendix VIII of this document.

Aquatic Ecology Laboratory

SOP 1.2 Fluorometric Analysis of Chlorophyll *a* Using Grinding

Development date – 2002	Author(s) – *Matthew McIver*
Revised – April 2014, Revision 3	Author(s) – *Matthew McIver*
Reference(s) – [1]EPA method 445.0 [2]Turner Designs 10-AU User's Manual [3]Welschmeyer, N.A. 1994. Fluorometric analysis of chlorophyll *a* in the presence of chlorophyll *b* and phaeopigments. Limnl. Oceanogr. 39:1985-1992.	

I. Introduction

A. *Purpose*: Chlorophyll *a* is analyzed in order to quantify phytoplankton biomass. **This method is indicated for samples that require analysis using NC DWQ certified methods.**

B. *Summary of Method:* A selected volume of water is filtered through glass fiber filters, concentrating the phytoplankton. The filters are frozen, immersed in acetone and then macerated with a tissue grinder. Freezing and grinding ruptures the cells, releasing the photosynthetic pigment chlorophyll *a* which dissolves in the acetone. The chlorophyll *a* in the acetone is quantified using a fluorometer with a specific set of filters and light source which eliminates the need for acidification to address spectral interference from pheophytin and chl *b*. This method requires the use of equations for "uncorrected chlorophyll *a*". The light source is a blue lamp, which passes through a filter that allows the wavelengths at 436 nm to excite the sample. The light emitted by this excitation then passes through a filter that allows wavelengths at 680 nm to pass on to a photo-multiplier which quantifies this light.

II. Supplies/Instruments

- 125 & 250 mL light-proof plastic bottles
- Filtration funnel – 25 mm base
- 50 mL graduated cylinder
- Vacuum pump with gauge
- 25 mm glass fiber filters (nominal pore size 0.7 micron)
- Forceps
- Aluminum foil
- Permanent marker
- Freezer capable of -20^{o}C
- Plastic airtight container with desiccant
- Certified thermometer for freezer
- Racks of 50 mL centrifuge tubes with screw caps, polypropylene
- Masking tape
- Arrow JR4000 overhead electric stirrer
- Kontes Potter-Elvehjem tissue grinder size 22 pestle
- 2-10 mL pipette with tips
- 90% acetone solution
- 100 mL beaker
- 100% acetone solution {VWR # BDH1101-19L}
- Waste acetone container
- Kimwipes {Fisher # 06-666A}
- Squirt bottles for DI water
- Turner 10-AU Fluorometer with filter set10-040R
- Fluorometer cuvettes

III. Sample Collection and Filtration

1. A chain of custody sheet (COC 1.2.1) should be filled out for each set of samples.
2. Samples should be collected in 125 mL light-proof bottles, a 250 mL bottle should be used for samples requiring bottle duplicates (10% of samples should be analyzed in duplicate). Fill bottle to about 95% full to allow for mixing before filtration.
3. Samples should be placed on ice or refrigerated until filtration. Filter samples as soon as possible; maximum holding time is 24 hours.
4. One aliquot is filtered for each sample. A bottle duplicate must be filtered once per ten samples.
   [During filtration the goal is to filter enough water to keep the chlorophyll a concentration below the UDL, thereby eliminating an extract dilution step, while allowing for enough to be analyzed accurately.]
5. Shake the sample for 10 seconds to mix, measure 20 mL in a graduated cylinder and pour into the filter funnel.
6. Apply suction not to exceed 6 in Hg (20kPa). As the sample leaves the funnel turn off the suction to prevent possible cell rupture and chl *a* loss.
7. Look on the filter for a visible golden-brown or green color. If there is no color visible, measure and filter another 20 mL. Repeat until color is seen.
8. If the chlorophyll *a* concentration is expected to be less than 100 μg/L, one 50 mL aliquot may be filtered.

9. Fold filters in half with filtered material inside and place in aluminum foil wrapper. Fold foil and label with a permanent marker including the station and volume filtered.
10. Rinse graduated cylinder and filter funnels with di H2O.
11. When all samples have been filtered, filter 50 mL of di H2O as a laboratory reagent blank (LRB).
12. Wrap the filters together in a big piece of foil and label the outside with place of collection, date and amount filtered. Place this in the freezer in an air-tight plastic container with anti-humidity material (desiccant).
13. The temperature of the freezer should be recorded once per 24 hours during storage on logsheet LS 1.2.1.
14. Clean the filter apparatus with a hot water rinse and finish with a DI water rinse.

IV. Extraction of Chlorophyll *a*

1. Maximum hold time for frozen filters is four weeks.
2. Check pipette accuracy before and after grinding by dispensing 5 mL of diH20 onto a weigh boat on the balance. If the weight is not within +/-10% of 5 g, pipette needs to be adjusted. Record weights on the bench sheet.
3. Fill a 100 mL beaker with 90% acetone. In subdued light in a vent hood, place a sample filter in the 50 mL centrifuge tube and add 5 mL of 90% acetone solution using a pipette (filter can be ripped into 4 pieces to make grinding easier). Slide the centrifuge tube under the pestle and turn the knob to initiate grinding. Grind using the mechanical grinder set-up at a setting of 6 or less, grinding the filter completely.
4. Placing the centrifuge tube underneath to catch the runoff, rinse the grinder and sides of centrifuge tube with another 5 mL of 90% acetone. Turn the grinder on and allow the acetone to rinse the pestle. If more than 5 mL is necessary for rinsing record total volume on the bench sheet.
5. Place the tube into a rack covered with tape. A masking tape label is placed adjacent to each row of tubes to indicate which samples are in that row. When the rack is full place it in a refrigerator at 4^{o}C.
6. Use 100% acetone to rinse the pestle, catch the used acetone into a waste container.
7. Steep samples for a minimum of 2 hours but no more than 24 hours. *DWQ asked us to remain consistent with steep times, therefore we attempt to read chla the same day they are ground. Longer steep times should be noted on the bench sheet.*

V. Fluorometric Analysis

Turn on the fluorometer at least 24 hours before reading samples to allow for warm up. The fluorometer must be calibrated bi-monthly. Check the calibration (after warm up) with the solid secondary standard. The fluorometer value must be within 10% of the actual value (see LS 1.7.1) or calibration is required (SOP 1.7). If calibration is not necessary, record the solid secondary standard values on the bench sheets.

1. Remove tube racks from the refrigerator, cover the tops of the tubes with a clipboard and shake vigorously. Centrifuge each tube at 1000 g for 5 minutes. Measure the temperature of the tubes with the infrared thermometer. If temperature is not approximately 70^{o}F then let samples sit for a few minutes to warm up to room temperature.
2. A Secondary Check Standard should be read at the beginning and end of each sample set. See AEL SOP 1.7 to make and analyze the standard.
3. To analyze a sample, pour the supernatant into the fluorometer cuvette. About one-half full is fine. Place the tube in the chamber and close the lid. Press the * button. This will range

the sample and average the readings over 10 seconds. Record the value when the display says "done". Record value to one decimal place, without rounding.

4. Rinse the cuvette with 100% acetone and drain between samples. Use 3 cuvettes and alternate them to speed up analysis. Rinse the centrifuge tubes by pouring the excess acetone into the acetone waster container. Use a squirt bottle full of DI water to blast the filter pellet out of the bottom of the tube and pour into the acetone waste container. Place in a separate rack.
5. If the fluorometer value is > 450 (90% of the Upper Detection Limit = 500), the sample must be diluted and re-analyzed.
   A. Print out WS 1.2.1 and fill out appropriate information during dilution procedure.
   B. Place 9 mL of 90% acetone into a clean centrifuge tube using a clean pipettor tip. Add 1 mL of the sample extract using another clean tip and invert several times to mix.
   C. Pour into a clean cuvette and read as usual.
   D. Be sure to add in Dilution Factor when calculating final Chlorophyll *a* concentration.
6. Clean the cuvettes after use by rinsing them 3 times with DI water.
7. Clean the centrifuge tubes by rinsing the tubes 3 times with DI water and invert in a rack to dry. Tubes should be placed in the acid bath occasionally to remove accumulated grunge.
8. Record the fluorometer values and other appropriate information on the chl *a* bench sheet (BS 1.2.1). Room temperature and sample temperature should be recorded on the bench sheet.

VI. Computation of Chlorophyll *a* Concentration

1. Chlorophyll *a* concentration in μg/L (ppb) is calculated using this formula:

$$C_{S,u} = \frac{\text{extract volume (mL) x DF}}{\text{sample volume (mL)}}$$

$C_{S,u}$ = uncorrected Chlorophyll *a* concentration in whole water sample

extract volume = volume of extraction acetone

DF = dilution factor (volume of extract/total volume)

sample volume = volume of whole water filtered

VII. Quality Assurance/Quality Control

1. LRB's are analyzed with each set of filters. An LRB consists of di H2O that is filtered the same as samples after all samples have been filtered. Elevated LRB values indicate contamination at some point during the procedure. If the LRB value is >10% of the analyte value, the samples are considered contaminated and should be discarded.

2. A Quality Control Standard (Secondary Check Standard) of a source other than the calibration standard is measured at the beginning and the end of a batch of samples. The measured value should be within 10% of the actual concentration, if not the fluorometer may need to be recalibrated. See SOP 1.7 Fluorometer Calibration for details on the Secondary Check Standard.

3. The linear dynamic range has been determined for the fluorometer and the upper detection limit (UDL) has been established using this data. If a sample reads above 90% the UDL the sample must be diluted and re-analyzed.

BS 1.2.1 Lower Cape Fear River Program Chlorophyll *a* Bench Sheet

month/year-				
Station	filter 1	filter 2		
M18				mL filtered -
				sample date-
M61				Grinding time/date -
				Reading time/date -
NC11				Room Temperature -
				Sample Temperature -
				Analyst's Signature -
90% acetone blank value-				Actual Chla solid stnd values Hi- Lo-
LRB blank value-				Chl a standard value begin * -
				Chl a standard value end *-
				*Check Standard value must be within 10% of actual value, if not the fluorometer may need calibration.
				Pippettor check weight before-
				Pippettor check weight after-

Aquatic Ecology Laboratory

SOP 2.10 Water Column Cholorophyll *a* Determination, Non Grinding

Analysis of water column chlorophyll a using fluorometry.

<table>
<tr><td>Development date – ?</td><td>Author(s) – Matthew McIver</td></tr>
<tr><td>Revised – March 30, 2010</td><td>Author(s) – Matthew McIver</td></tr>
<tr><td colspan="2">Reference(s) –
Turner Designs. 1993. Model 10-AU-000 Laboratory Fluorometer User’s Manual. Part Number 10-AU-070. 845 W. Maude Avenue, Sunnyvale, CA 94086.

Welschmeyer, N.A. 1994. Fluorometric analysis of chlorophyll a in the presence of chlorophyll b and phaeopigments. Limnl. Oceanogr. 39:1985-1992.

U.S. Environmental Protection Agency. 1997. Methods for the Determination of Chemical Substances in Marine and Estuarine Environmental Matrices. Second Edition. EPA/600/R-97/072. U.S. Environmental Protection Agency. Cincinnati, Ohio.</td></tr>
</table>

I. Introduction

Chlorophyll *a* is analyzed as a quantification of phytoplankton biomass. This method employs the use of a specific set of filters which minimizes the interference from chl b and phaeopigments while removing errors inherent in the acidification procedure.

II. Supplies/Instruments

- Light-proof 120mL brown Nalgene sample bottles
- Di H2O
- 90% acetone
- 15 mL screw cap plastic (polypropylene) centrifuge tubes with racks
- Forceps
- Aluminum foil
- 3 funnel filtration manifold
- 25 mm glass fiber filters, nominal pore size of 0.7 micron

III. Sample Collection and Preparation

1. Samples should be collected in light-proof bottles in triplicate and stored on ice or refrigerated until filtered.
2. Filter samples as soon as possible; maximum holding time is 24 hours.
3. Using the 3 funnel manifold, simultaneously filter 50 ml (volume may vary depending on turbidity and algal concentration) from each bottle.
4. Invert sample several times to mix, measure volume in a graduated cylinder and pour into filter funnel.

5. Apply vacuum, pressure should not exceed 6 in Hg (20kPa). Turn off the vacuum as the sample leaves the funnel; **allowing vacuum on a dry filter may result in chlorophyll loss**.
6. A laboratory reagent blank (LRB) is made by filtering 50 ml of distilled water. This blank filter is treated like the sample filters.
7. Fold filters in half, filtered material on the inside and place in aluminum foil. Fold foil and label with a permanent marker, the station, date and amount filtered. Filters should be placed in a container with dessicant and kept in the freezer until analysis.
8. If analyzing nutrients from the filtrate, return about 70 ml of filtrate to a rinsed sample bottle (already labeled) for analysis of dissolved inorganic nitrogen and/or phosphorous. These samples should be kept in the freezer.
9. Clean filter manifold by flushing with DI water. A monthly 1% HCl rinse is recommended to further clean the apparatus.

IV. **Extraction of Chlorophyll *a***

1. Remove foil-wrapped filters from freezer. In subdued light, un-wrap filters, placing each filter in a 15 ml centrifuge tube with 10 ml of 90% acetone solution.
2. Place tube in a pre-labeled rack. The rack for chla tubes has pvc tubes cut to length to cover each tube to prevent light from entering the tube.
3. When the set is done or rack is full, place rack in the refrigerator.
4. Steep for a minimum of 18 hours but no more than 24 hours.
5. Remember - chlorophyll *a* is sensitive to heat and light, so minimize each!

V. **Fluorometric Analysis**

1. Turn on fluorometer and allow a 30 minute warm up.
2. Remove samples from refrigerator, **gently invert each tube several times** and return to tube rack. Place rack in the dark and allow to warm to room temperature.
3. Pour acetone from tube into the fluorometer cuvette, about 1/2 full is fine. Place tube in chamber and close lid. Allow several seconds for the fluorometer to determine and set the range appropriate for the sample concentration. Machine will beep until this is finished.
4. Press the * button. This will average the readings over 10 seconds. Record the value when display says, "done". *If the fluorometer displays 'over' the sample concentration is above the maximum level that the instrument can read (500) and must be diluted. Put 9 ml of 90% acetone in a clean cent tube and pipette 1ml of sample into tube. Invert and read on fluorometer as above. Raw fluorescence number should be multiplied by 10.*
5. Pour extract back into centrifuge tube and place in another rack. Rinse cuvette with 100% acetone and drain between samples. Use 3 cuvettes and alternate them to speed up analysis.
6. To clean centrifuge tubes - pour excess extract into acetone waste receptacle. Remove filter from tube and discard. Rinse twice with tap water and once with DI water. Place inverted in rack to dry. Tubes should be acid-washed occasionally to remove built up residue.

VI. **Computation of Chlorophyll *a***

1. If the LRB fluorometer value is 1.0 or greater, subtract this value from the fluorometer value before Chl *a* calculation.
2. For chlorophyll *a* concentration in µg/liter (parts per billion), multiply the flourometer reading by this factor:

   ml 90% acetone added / ml of sample water filtered, e.g. 10/75 = 0.133 or 10/50 = 0.200

3. Some of our projects have chla report sheets which will automatically calculate chla concentration when the raw numbers are entered. The mean and standard deviation for the replicate samples is also calculated and they are printed out in a form to be placed in respective data folders.

Lower Cape Fear River Program - Chlorophyll *a* Analysis (Non Grinding) 1

UNCW Aquatic Ecology Laboratory

month/year-

Station	replicate 1	replicate 2	replicate 3		
NAV					Dropper Initials/Time/Date-
HB					mL filtered-
BRR					Analyst's Initials/Time/Date
M61					90% acetone blank value-
M54					DI blank value-
M35					Analyst's Signature -
M23					Sample Temperature -
M18					

Station	replicate 1	replicate 2	replicate 3		Dropper Initials/Time/Date-
6RC					mL filtered-
LCO					Analyst's Initials/Time/Date
GCO					90% acetone blank value-
SR					DI blank value-
BRN					Analyst's Signature -
HAM					Sample Temperature -

Station	replicate 1	replicate 2	replicate 3		Dropper Initials/Time/Date-
NCF117					mL filtered-
B210					Analyst's Initials/Time/Date
COL					90% acetone blank value-
SR-WC					DI blank value-
LVC2					Analyst's Signature -
SC-CH					Sample Temperature -

Aquatic Ecology Laboratory

SOP 2.2 Biochemical Oxygen Demand, Natural Surface Waters, 5 and 20 day

Development date – 1996	Author(s) – *Scott Ensign, Matthew McIver*
Revised – January 7, 2010	Author(s) – *Matthew McIver*
Reference(s) – Standard Methods 20th edition, 5210 B	

I. Introduction

Purpose: Bacterial respiration requires oxygen and in natural surface waters it is beneficial to quantify this oxygen demand. Organic material introduced to surface waters can often be problematic in that the resultant increase in oxygen demand may lead to oxygen depletion: a stressful event for aquatic organisms. There are also several chemical processes in surface waters that will result in oxygen demand, thus the name "Biochemical Oxygen Demand" or BOD.

Summary of Method: To quantify BOD water is collected from location of interest and transported to the lab. The water is aerated such that saturation is achieved. The water is then placed in special BOD bottles and the dissolved oxygen measured. The bottles are placed in an incubator with constant temperature (20^{o}C) for a set number of days and then the dissolved oxygen is measured again. The amount of oxygen used is calculated through subtraction and this number is the BOD value. A standard of 5 or 20 days is typically used.

II. Supplies/Instruments

- One liter plastic bottles
- 300 ml glass BOD bottles, stoppers and caps
- Coated metal BOD bottle racks
- 1 liter graduated cylinder
- plastic wand with perforated Plexiglas disk (see attached photo)
- incubator set at 20^{o} Celsius (+/- 1^{o})
- Dissolved oxygen meter with BOD probe
- Glass thermometer
- pH meter
- *Contrad 70* laboratory soap mixed 2% v/v (2ml per 100 ml of DiH2O)
- Bleach
- DI H20
- BOD bench sheet
- Kimwipes
- Barometer

- YSI 5000 Calibration Log
- 3 M KCl
- pH 4.0 buffer solution
- pH 7.0 buffer solution

III. Sample Collection and Preparation

1. Collect samples in one liter plastic bottles. Dirty bottles are cleaned by soaking in a 10% HCl bath for 24 hours, then rinsing and drying. It is always advisable to collect water directly in-stream, but a thoroughly rinsed bucket may be used. Samples are kept on ice and in the dark until time of setup. Samples should be run within 24 hours of collection.
2. Raise temperature of samples to 20^{o} C by soaking in warm water, measuring temperature with glass thermometer. Store samples in the incubator until ready to set up.
3. Place glass BOD bottles in rack and record bottle numbers on BOD data sheet.
4. For each sample, pour 750 mL of sample into a 1000 mL graduated cylinder and aerate for five seconds with aeration wand. This allows the dissolved oxygen saturation to reach near 100% by either releasing oxygen from super-saturated samples or adding oxygen to samples below saturation. *If air bubbles develop in the tops of the bottles during incubation it is likely because this step was not performed correctly.*
5. Fill the 300 ml BOD bottles to the top with sample water and let bottles rest for 30–60 minutes. Duplicates are run for each sample location.
6. A distilled water blank should be run in sequence at the beginning of the bottle rack and at the end. A BOD of >0.2 in a blank indicates some interference, possibly a result of incomplete cleaning of the DO probe between samples.

IV. Dissolved Oxygen Meter Air Calibration

8. Stopper BOD bottle used for calibration and shake vigorously to saturate the air inside. Gently dab membrane on BOD oxygen probe and place in calibration bottle.
9. Using the menu at the bottom of the screen, select “Calibrate”, then “DO Cal”. Select “Next” twice until salinity is blinking, adjust salinity to 0.0. Select “Next” once and barometric pressure will blink. Enter the correct barometric pressure using lab barometer. Press the enter button and the values will be saved and instrument will return to calibration mode.
10. Allow probe 15 minutes to warm-up and equilibrate. *This is a good time to measure pH*.
11. When the DO is stable select “Auto Cal.”
12. DO is now calibrated, return to main display by pressing “Mode”.
13. DO probe membranes should be replaced every other month. Record date of change in YSI Calibration Log.

V. pH Meter Calibration and Measurement (Fisher Scientific Accumet Basic)

*Initial, 5 day and 20 day pH should be measured for each sample.

1. Plug the meter in to turn it on. Press pH/mV button until pH reads on the screen. We calibrate the meter at 7.0 and 4.0 since this brackets most of our sample pH values.
2. Press ‘Setup’ and “Clear Buffers” will blink, press ‘Enter’. The meter will go back to pH display and show “Measurement” at the bottom. Open tab at side/top of pH probe. Immerse probe tip in 7.00 pH buffer and when ‘S’ is constantly displayed in the upper left of the display press “Standardize”.
3. Rinse probe with distilled water and dab with a kimwipe.
4. Immerse probe tip in 4.00 buffer and when “S” is displayed, press “Standardize”, display will show a diagnostic and state if the probe is good, then switch back to measurement mode.

5. Wash and dab the probe and place in each BOD, recording sample pH on data sheet.
6. To store probe, close the tab and place in 3M KCl. Do not use pH buffer.

VI. Initial DO Measurement

1. To measure DO in each bottle place the BOD probe into the bottle and turn on the stirrer. Any air bubbles adhering to the sides of the bottle and/or the probe will spin up towards the surface. Raise probe slightly to allow these bubbles to escape.
2. Allow ~30 seconds for DO value to equilibrate. **[If the initial DO is > 9.0, the sample is supersaturated and air bubbles will form in the BOD bottles over the 5 day incubation period. Return sample to Grad Cylinder and swoosh.]** Record DO value on the bench sheet (value is recorded to the tenth place). Rinse and dab the probe between each station.
3. Each bottle should be checked for air bubbles on the sides of the bottle. Use a plastic stopper to tap on the bottle and force the bubbles to rise to the top.
4. Stopper and cap each bottle. Extra water in the bottle will form water seal above the stopper in the neck of the bottle, which the plastic cap will keep from evaporating. A little di water can be added to get a good water seal.
5. Place in incubator at 20^0 C.

VII. BOD5 and BOD20 Readings

1. Calibrate DO and pH meters as described above.
2. Record incubator temperature on bench sheet (use certified thermometer).
3. Measure DO and pH of each sample, rinsing probe and dabbing dry between samples.
4. Record values in tenths on bench sheet.
5. If performing 5 day BOD measurement, re-seal and cap bottles and place back in incubator for BOD20 incubation. Ensure no air bubbles are trapped in the bottle after the stopper is replaced.
6. After 20 day BOD has been measured, the bottles are emptied and rinsed two times with hot tapwater. Place about 100 ml of lab soap solution into bottle, shake for 10 seconds, rinse with di water and allow to air dry for storage. The BOD bottle storage box is labeled for each bottle by number.

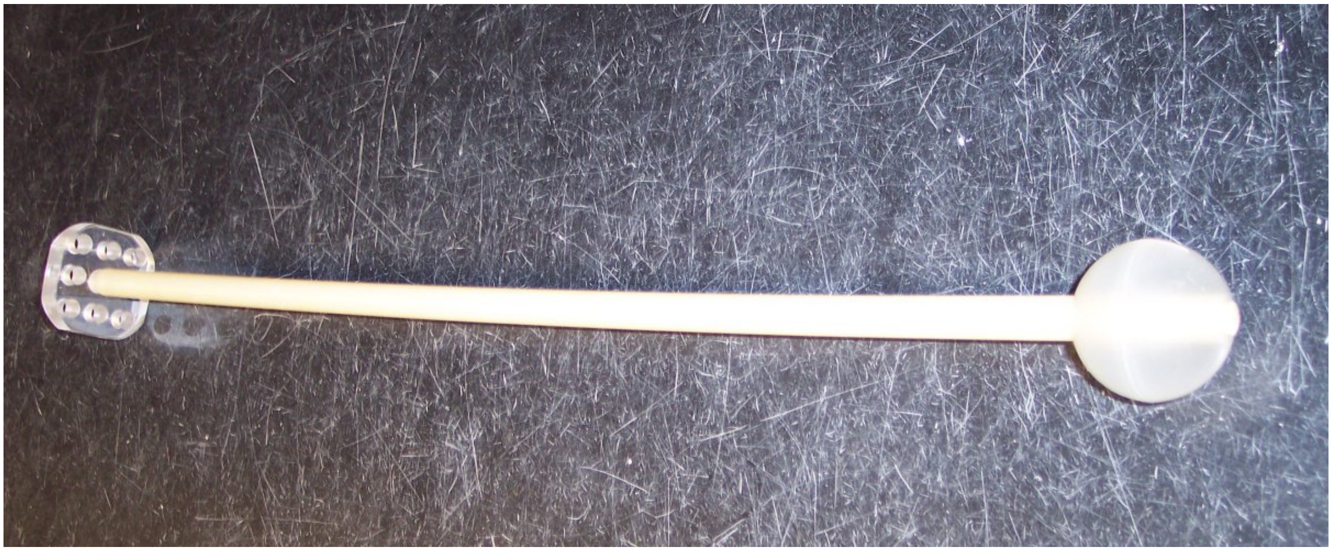
*If the BOD5 value is at or above 5, the sample must be diluted by 50% before being placed back in the incubator. Previous experience shows that a sample that has a BOD value of 5 after the fifth day will use all its available oxygen before day 20. Therefore, without dilution, the 20 day reading will be inaccurate.

VIII. References

American Public Health Association. Standard Methods for the Examination of Water and Wastewater 20th Ed. 1998. Pg 5-2.

NCDEHNR. 1993. Long-Term Biochemical Oxygen Demand Procedure. North Carolina Department of Environment, Health, and Natural Resources, Division of Environmental Management. Raleigh, N.C.

NCDEHNR. Five Day BOD Test Procedure (Bottle Method Technique). North Carolina Department of Environment, Health, and Natural Resources, Division of Environmental Management. Raleigh, N.C.



Typical BOD Bench Sheet

BOD Bench Sheet - Lower Cape Fear River Program River Boat Trip														
Collection				Set Up			5 Day Reading			20 Day Reading				
Scientist:				Analyst:			Analyst:			Analyst:				
Day:				Day:			Day:			Day:				
Date:				Date:			Date:			Date:				
Time:				Time:			Time:			Time:				
				Incubator Temp:			Incubator Temp:			Incubator Temp:				
Station	**Bottle #**	**Field pH**		**Setup pH**	**Initial D.O.**		**5 day pH**	**5 day DO**		**20 day pH**	**20 day DO**		**BOD 5 Initial minus 5 day**	**BOD 20 Initial minus 20 day**
BLANK		na		na			na			na				
NC11														
AC														
BBT														
BLANK		na		na			na			na				

Section 10 - *Data Management and Data Quality*

An integral part of any research program is the proper handling of data. This section describes data flow and quality control procedures used by the AEL with regards to Lower Cape Fear River Program data.

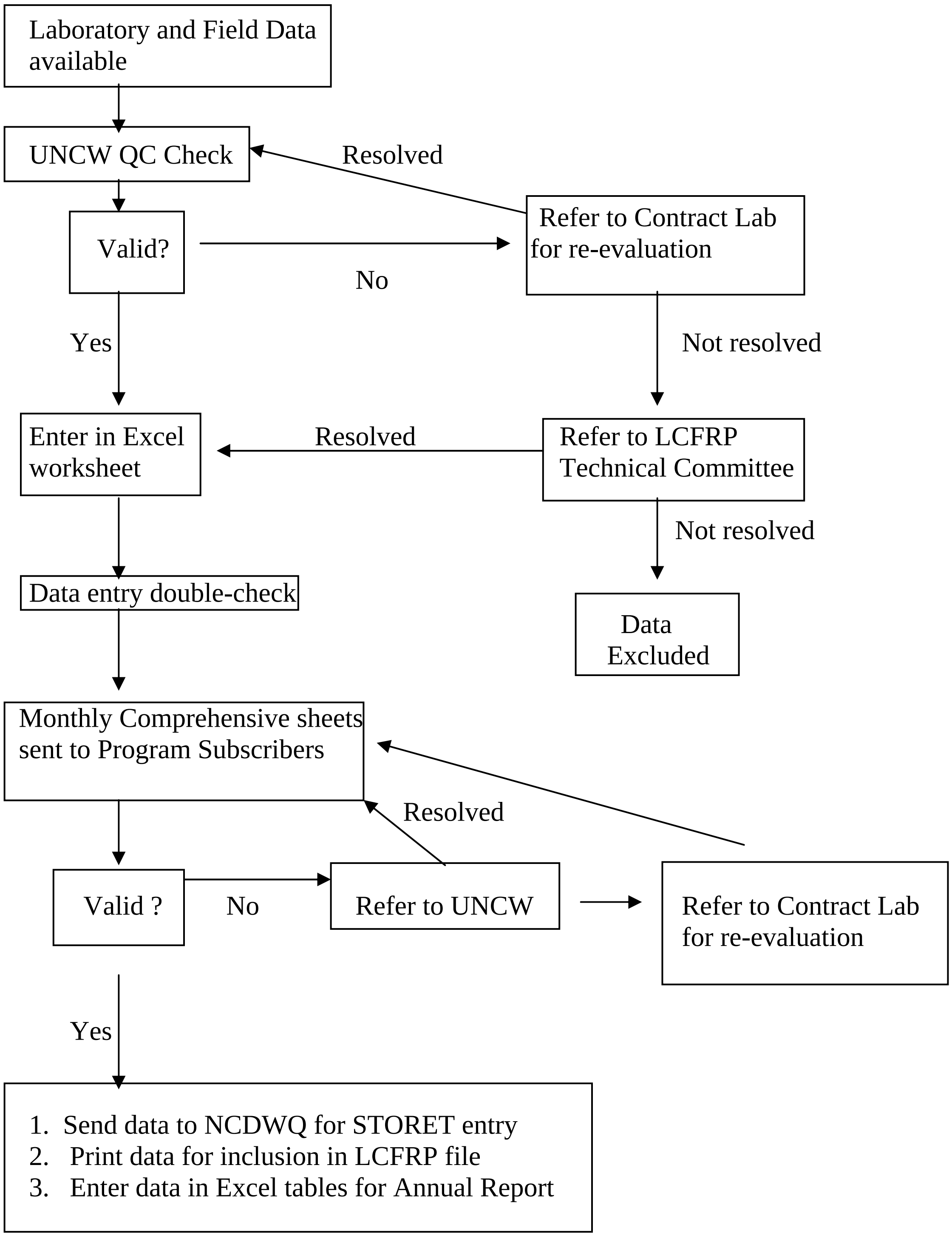
Data Management

LCFRP data from the state certified contract laboratory is sent to the Program Research Director. When the Program Research Director accepts the data as valid, data required per the Memorandum of Agreement is entered into Microsoft Excel spreadsheets. The Excel spreadsheet is checked for data entry errors and the final copy is reviewed by the Program Sampling Coordinator. The data are then sent to each NPDES Subscriber of the LCFRP and other interested parties. Questions about the data are referred to the Program Research Director for re-evaluation or clarification which may include re-analysis by the contract laboratory. If no questions arise the data are then sent to the NC Division of Water Resources Coalition Coordinator (NCDWR) for entry into the EPA STORET system. See page 2, this section for data flow chart.

A data status sheet is checked to ensure that data is sent to the proper entities on a timely basis (see page 3). A data exclusion sheet will be maintained at the Aquatic Ecology Laboratory for the purpose of reporting and explaining MOA required samples that were not collected (see page 4). Any data exclusions will be reported and explained in the annual LCFRP report to NCDWR per the MOA.

A copy of all data is kept on file in the UNCW Aquatic Ecology Laboratory. An annual report detailing findings during the period from January to December is written by the scientists each year (see Section 12).

Lower Cape Fear River Program
Data Flow Sheet



Lower Cape Fear River Program Data Status (please initial and date each entry) **2006-2007**

Month of Collection	Sampling Trip	Date Sampled	Entered on EXCEL	EXCEL Doublecheck	EXCEL sheets to LCFRP Subscribers (<45 days after collection)	Data presented to Technical committee	Data E-mailed to DWQ (<90 days after end of collection month)
October	Black River Watershed						
	Cape Fear Estuary						
	Cape Fear Riverine						
	NECFR Watershed						
November	Black River Watershed						
	Cape Fear Estuary						
	Cape Fear Riverine						
	NECFR Watershed						
December	Black River Watershed						
	Cape Fear Estuary						
	Cape Fear Riverine						
	NECFR Watershed						
January	Black River Watershed						
	Cape Fear Estuary						
	Cape Fear Riverine						
	NECFR Watershed						
February	Black River Watershed						
	Cape Fear Estuary						
	Cape Fear Riverine						
	NECFR Watershed						
March	Black River Watershed						
	Cape Fear Estuary						
	Cape Fear Riverine						
	NECFR Watershed						
April	Black River Watershed						
	Cape Fear Estuary						
	Cape Fear Riverine						
	NECFR Watershed						

Lower Cape Fear River Program Data Exclusion Sheet

Month	Station	Data Excluded	Reason for Data Exclusion

Data Quality Objectives

- **Completeness**: **It is imperative that all data is collected per schedule in the MOA (Appendix I) and analyzed to the levels dictated by the MOA**. Resampling may be necessary because of lost samples, instruments out of calibration, etc. If conditions prevent sampling notify DWR coalition coordinator within one week.

- **Representativeness**: The Lower Basin of the Cape Fear River is a diverse system composed of riverine and estuarine habitats. Monitoring sites for this study were selected to adequately represent these regions. The program uses sampling techniques developed for riverine and estuarine areas.

- **Comparability**: All Lower Cape Fear River personnel follow the same protocols when collecting samples. Standard Operating Procedures for sample collection are described in detail in Section 6 of this document. This consistency in methodology ensures comparability of the data.

- **Quality Control**: The use of laboratory blanks, field duplicates, lab control samples, matrix spikes and duplicates, and compound identification and quantification are components of the Quality Control methods used by the state certified contract lab. The Quality Control procedures used by the contracted state-certified laboratory are described for each method used to analyze samples. A list of methods used by the state certified contract laboratory can be found in Appendix VIII.

- **Precision**: Analytical precision is evaluated by analysis of laboratory quality control samples such as duplicate control samples, matrix spike duplicates, and sample duplicates. Because the river is a flowing system, duplicate field measurements do not sample the same volume of water. Duplicate field measurements do not always agree.

- **Accuracy**: Analytical accuracy is evaluated by analysis of laboratory quality control samples, surrogate standard matrix samples, initial and continued calibration of equipment and blind samples. For this project, field accuracy is assessed using standard calibration techniques before and immediately following each sampling trip.

Section 11 - *QA/QC Audits*

Annual internal audits of both field and laboratory portions of the Lower Cape Fear River Program will be conducted by members of the LCFRP.

The audits will consist of a critical review of:

- Field data sheets
- Chain of custody sheets
- Laboratory log sheets
- Calibration worksheets
- Sample hold times
- Lab bench sheets
- Thermometer and reagent expiration dates
- Sample check in procedures

There are no formal audit forms, therefore the auditors should review SOPs (Appendix VIII) and have analysts perform a mock analysis. During the mock analysis the auditor should review each step of the SOP and verify that all steps are performed and that all QA/QC procedures are documented.

Appendix I - Memorandum of Agreement

Memorandum of Agreement
Between
The State of North Carolina's Division of Water Quality
And
The Lower Cape Fear River Program (LCFRP) Permittees

Effective:
July 1, 2011 through June 30, 2016

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MEMORANDUM OF AGREEMENT

This AGREEMENT ("Agreement") is made by and between the DIVISION OF WATER QUALITY, NORTH CAROLINA DEPARTMENT OF ENVIRONMENT AND NATURAL RESOURCES, or its successor agency or department ("DWQ"), and the NPDES Dischargers who have voluntarily approved this Agreement, known and referenced collectively as the "PERMITTEES". The Agreement includes all the attached tables and appendices.

The purpose of this Agreement is to establish a formal agreement between DWQ and the PERMITTEES whose responsibilities include surface water monitoring and reporting within the Lower Cape Fear River Basin below Lock and Dam #1 to monitor strategically located surface water sites and parameters to obtain information on water quality in the basin. PERMITTEES shall subcontract with one organization to collect instream monitoring data normally collected by the individual permittees. Monitoring sites and parameters have been established by the PERMITTEES and DWQ such that the instream monitoring is more efficient, effective, flexible, and basin-oriented.

The PERMITTEES who are participating in this Agreement, listed in Table 1, are exempted from any instream monitoring as specified in their individual NPDES permits beginning on the effective date of this Agreement and continuing for the duration of each permittee's participation in this agreement. This Agreement does not affect any influent or effluent monitoring requirement or any other of the NPDES permit requirements of individual permit holders with the one exception of performing upstream and downstream water quality monitoring. Subsequent to the execution of this Agreement, the DWQ will issue a letter to each permittee notifying the permittee that the instream monitoring requirements of its permit are not effective for as long as this Agreement is in place and the permittee remains a party to this Agreement.

The PERMITTEES will contract for the performance of the monitoring activities described herein with a DWQ-certified contract lab, organization, or agency, who shall subcontract, as necessary, with a laboratory appropriately certified by DWQ for the required analysis. Sample collection and field measurements will be made by the PERMITTEES, the contractee or a sub-contractee who will act as agent(s) of the PERMITTEES. Each permittee has the right to review and comment on work, data or reports prepared by any contractee on behalf of the PERMITTEES prior to its submission to DWQ and to notify DWQ of any objection or disagreement with any portion of the work, data, or reports. Unless such notice is made within thirty (30) days of submission to DWQ, it shall be deemed to be waived and the work, data and reports submitted shall be deemed to be approved by the PERMITTEES. It will be the responsibility of the PERMITTEES or their contractee to coordinate the collection and analyses of the water quality monitoring data at the locations, parameters, and frequencies specified in Appendix A. Sample collection and field measurement methods shall follow procedures outlined in Appendix B. The PERMITTEES or their contractor shall submit the water quality data to DWQ using the format described in Appendix C, preferably Microsoft Excel 2000 or a subsequent version, or the equivalent. The Permittees or their contractor shall submit the water quality data to DWQ within 90 days of the end of the month in which the sampling was performed. All data sheets shall be archived by the PERMITTEES or their

contractee for a period of 5 years.

PERMITTEES, or their contractee shall submit an annual written report of its compliance or non-compliance with the monitoring requirements as specified within this Agreement no later than April 30th each year that this Agreement is in effect. The report shall include the NPDES permit number of each actively participating permittee, the cause of any non-compliance with stipulations of the Agreement, any remedial action taken and the probability of meeting the next schedule requirements. Additional requirements for the reports are outlined in Appendix C. Two signed copies of these and any other reports required herein, shall be submitted to the DWQ Coalition Coordinator(s) at1621 Mail Service Center Raleigh NC 27699-1621.

Failure by PERMITTEES or their contractee to collect the water quality data as described in this Agreement or to provide the data to DWQ in the required format may result in the revocation of this Agreement by DWQ and the return to individual upstream and downstream monitoring requirements, as specified in individual NPDES permits of the PERMITTEES.

Stream sampling may be discontinued at such times as flow conditions in the receiving waters or extreme weather conditions will result in a substantial risk of injury or death to persons collecting samples. Sampling may also be discontinued when environmental conditions, such as a dry stream, prevent sample collection. In such cases, for each day that sampling is discontinued, DWQ Coalition Coordinator(s) shall be notified within one week of the discontinuance and written justification for the discontinuance shall be submitted with the monthly data submittal. This provision shall be strictly construed and may not be utilized to avoid the requirements of this Agreement when performance of these requirements is attainable. When there is a discontinuance pursuant to this provision, sampling shall be resumed at the first opportunity after the risk period has ceased.

This Agreement may be modified by written consent of both parties. DWQ or the PERMITEES may determine that it is necessary to request changes in monitoring frequency, parameters or sites to be sampled. Any such changes can only be made by a written amendment to this Agreement agreed to by DWQ and a majority of the PERMITTEES then parties to the Agreement. The amendment shall be signed by PERMITTEES' primary contact and by DWQ. Such amendments may be entered into at any time.

The parties may also desire to allow Dischargers 1) who, subsequent to the date of this Agreement, receive NPDES permits within the Lower Cape Fear River Basin or 2) who have NPDES permits within the Lower Cape Fear River Basin but are not parties to this Agreement to enter into this Agreement subsequent to the effective date hereof. Any such changes can only be made by a written amendment to this Agreement agreed to by DWQ and a majority of the PERMITTEES then party to the Agreement. The amendment shall be signed by PERMITEES' primary contact and by DWQ and, if appropriate, by an authorized officer of any such Discharger who wishes to enter into the Agreement subsequent to the effective date hereof. DWQ will consider modification of existing monitoring requirements for any such discharger similar to those in effect for the existing PERMITTEES. Such amendments may be made at any time that this Amendment is in effect.

This Agreement shall be effective until June 30, 2016 unless extended by the consent of both parties. Upon 60 days written notice, DWQ or a majority of the PERMITTEES then party to the Agreement may terminate this Agreement for any reason. Upon termination of this Agreement, the monitoring requirements contained in the individual NPDES permits of the PERMITTEES shall become effective immediately.

An individual permittee may terminate and cancel its participation in this Agreement by providing 60 days written notice to the PERMITTEES, the DWQ Coalition Coordinator(s), the appropriate DWQ Regional Office, and the DWQ NPDES Unit. The monitoring requirements contained in the individual NPDES permit shall become effective immediately upon such cancellation or termination. In the event a permit holder terminates or cancels its participation in this Agreement, the PERMITTEES may request that DWQ review the monitoring plan described in this Agreement for a possible reduction in sampling effort or requirements.

IN WITNESS WHEREOF, the parties have caused the execution of this instrument by authority duly given, to be effective as of the date executed by the DWQ.

DIVISION OF WATER QUALITY	**LOWER CAPE FEAR RIVER PROGRAM**
By: ____________	By: ____________
Coleen Sullins **Director** **Division of Water Quality**	**Chris May** **Chairman** **Lower Cape Fear River Program**
Date: 6/30/11	Date: June 17, 2011

LCFRP PERMITTEE SIGNATURES

Permittee	NPDES Number	Signature
DAK Americas, LLC	NC0000663	Penny Mahoney Operating Director
Invista S.áR.L.	NC0001112	Rick Bayless Environmental Health and Safety Manager
Global Nuclear Fuels Americas	NC0001228	Shawn O'Connor Environmental Specialist
Carolina Power and Light (CP&L) d/b/a Progress Energy Carolinas, Inc. Sutton Steam Electric Plant	NC0001422	Mark Frederick Plant Manager
International Paper Company Riegelwood Mill	NC0003298	Edward Kreul Manager - Environment, Health, Safety, and Sustainability

LCFRP PERMITTEE SIGNATURES

Permittee	NPDES Number	Signature
DAK Americas, LLC	NC0000663	Penny Mahoney Operating Director
Invista S.áR.L.	NC0001112	Bill King - Authorized Signatory ~~Rick Bayless~~ BILL KING ~~Environmental Health and~~ Safety ~~Manager~~ SITE MANAGER
Global Nuclear Fuels Americas	NC0001228	Shawn O'Connor Environmental Specialist
Carolina Power and Light (CP&L) d/b/a Progress Energy Carolinas, Inc. Sutton Steam Electric Plant	NC0001422	Mark Frederick Plant Manager
International Paper Company Riegelwood Mill	NC0003298	Edward Kreul Manager - Environment, Health, Safety, and Sustainability

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DAK Americas, LLC	NC0000663	Penny Mahoney Operating Director
Invista S.áR.L.	NC0001112	Rick Bayless Environmental Health and Safety Manager
Global Nuclear Fuels Americas	NC0001228	for. N.K. Holmes Shawn O'Connor Environmental Specialist
Carolina Power and Light (CP&L) d/b/a Progress Energy Carolinas, Inc. Sutton Steam Electric Plant	NC0001422	Mark Frederick Plant Manager
International Paper Company Riegelwood Mill	NC0003298	Edward Kreul Manager - Environment, Health, Safety, and Sustainability

RECEIVED
JUN 10 2011
Environmental Sciences Section

LCFRP PERMITTEE SIGNATURES

Permittee	NPDES Number	Signature
DAK Americas, LLC	NC0000663	Penny Mahoney Operating Director
Invista S.áR.L.	NC0001112	Rick Bayless Environmental Health and Safety Manager
Global Nuclear Fuels Americas	NC0001228	Shawn O'Connor Environmental Specialist
Carolina Power and Light (CP&L) d/b/a Progress Energy Carolinas, Inc. Sutton Steam Electric Plant	NC0001422	J. Mark Frederick Mark Frederick Plant Manager
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International Paper Company Riegelwood Mill	NC0003298	Edward Kreul Manager - Environment, Health, Safety, and Sustainability

Momentive Specialty Chemicals	NC0003395	~~April Hanson~~ RONALD BAZINET ~~Environmental Engineer~~ SITE LEADER
Elementis Chromium LP	NC0003875	Joel Barnhart Vice President, Technical
Town of Mount Olive Mt. Olive WWTP	NC0020575	Charles Brown Town Manager
Town of Burgaw Burgaw WWTP	NC0021113	Kenneth T. Cowan Mayor
Town of Warsaw Warsaw WWTP	NC0021903	J. R. Steigerwald Town Manager
Town of Carolina Beach Carolina Beach WWTP	NC0023256	Tim Owens Town Manager
Cape Fear Public Utility Authority Northside WWTP	NC0023965	Matthew W. Jordan General Manager

Momentive Specialty Chemicals	NC0003395	April Hanson Environmental Engineer
Elementis Chromium LP	NC0003875	Joel Barnhart Vice President, Technical
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Elementis Chromium LP	NC0003875	______________________ Joel Barnhart Vice President, Technical
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Town of Carolina Beach Carolina Beach WWTP	NC0023256	______________________ Tim Owens Town Manager
Cape Fear Public Utility Authority Northside WWTP	NC0023965	______________________ Matthew W. Jordan General Manager

Momentive Specialty Chemicals	NC0003395	____________________ Ronald Bazinet Site Leader
Elementis Chromium LP	NC0003875	____________________ Joel Barnhart Vice President, Technical
Town of Mount Olive Mt. Olive WWTP	NC0020575	____________________ Charles Brown Town Manager
Town of Burgaw Burgaw WWTP	NC0021113	____________________ Kenneth T. Cowan Mayor
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Cape Fear Public Utility Authority Northside WWTP	NC0023965	Matthew W. Jordan ________________________ Matthew W. Jordan General Manager

Cape Fear Public Utility Authority Southside WWTP	NC0023973	Matthew W. Jordan General Manager
Town of Beulaville Beulaville WWTP	NC0026018	Kenneth Smith Mayor
Cape Fear Public Utility Authority Walnut Hills Subdiv. WWTP	NC0039527	Matthew W. Jordan General Manager
Brunswick Regional Water & Sewer H2GO Belville WWTP	NC0075540	Carl Antos Chairman
Brunswick County NE Brunswick Regional WWTP	NC0086819	Marty Lawing County Manager

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Brunswick County NE Brunswick Regional WWTP	NC0086819	________________ Marty Lawing County Manager

Table 1. LCFRP Permittees

NPDES Permit Number	LCFRP Permittees Ownership and Facility	Authorized Representative and Title	County	Region	Sub Basin	8 Digit HUC
NC0000663	DAK Americas, LLC	Penny Mahoney Operating Director	Brunswick	Wilmington	030617	03030005
NC0001112	Invista S.á R.L.	Rick Bayless Environmental Health and Safety Manager	New Hanover	Wilmington	030617	03030005
NC0001228	Global Nuclear Fuels – Americas	Shawn O'Connor Environmental Specialist	New Hanover	Wilmington	030617	03030007
NC0001422	Carolina Power and Light (CP&L) d/b/a Progress Energy Carolinas, Inc. Sutton Steam Electric Plant	Mark Frederick Plant Manager	New Hanover	Wilmington	030617	03030005
NC0003298	International Paper Company Riegelwood Mill	Edward Kreul Manager – Environment, Health, Safety, and Sustainability	Columbus	Wilmington	030617	03030005
NC0003395	Momentive Specialty Chemicals	April Hanson Environmental Engineer	Columbus	Wilmington	030617	03030005
NC0003875	Elementis Chromium LP	Joel Barnhart Vice President, Technical	New Hanover	Wilmington	030623	03030007
NC0020575	Town of Mount Olive Mt. Olive WWTP	Charles Brown Town Manager	Wayne	Washington	030621	03030007
NC0021113	Town of Burgaw Burgaw WWTP	Kenneth Cowan Mayor	Pender	Wilmington	030623	03030007
NC0021903	Town of Warsaw Warsaw WWTP	J. R. Steigerwald Town Manager	Duplin	Wilmington	030619	03030006
NC0023256	Town of Carolina Beach Carolina Beach WWTP	Tim Owens Town Manager	New Hanover	Wilmington	030617	03030005
NC0023965	Cape Fear Public Utility Authority Northside WWTP	Matthew W. Jordan General Manager	New Hanover	Wilmington	030617	03030005
NC0023973	Cape Fear Public Utility Authority Southside WWTP	Matthew W. Jordan General Manager	New Hanover	Wilmington	030617	03030005
NC0026018	Town of Beulaville BeulavilleWWTP	Kenneth Smith Mayor	Duplin	Wilmington	030622	03030007
NC0039527	Cape Fear Public Utility Authority Walnut Hills Subdivision WWTP	Matthew W. Jordan General Manager	New Hanover	Wilmington	030617	03030007
NC0075540	Brunswick Regional Water & Sewer H2GO Belville WWTP	Carl Antos Chairman	Brunswick	Wilmington	030617	03030005
NC0086819	Brunswick County NE Brunswick Regional WWTP	Marty Lawing County Manager	Brunswick	Wilmington	030617	03030005

APPENDIX A – LCFRP MONITORING PLAN

Table A-1 LCFRP Sampling Stations, Parameters and Sampling Frequency

Station Number	LCFRP Station ID	Location Description	Station Comments	Latitude (dd.dddd)	Longitude (dd.dddd)	County	Region	Index	8 Digit HUC	Stream Class	[1]Field Measurements	[2]Nutrients	[3]Metals	Lab Turbidity	TSS	Chloro-phyll *a*	Entercocci	Fecal Coliform
B8340050	BRN	Browns Creek at NC87 nr Elizabethtown	hog farm area	34.6136	-78.5848	Bladen	FRO	18-45	03030005	C	M	M		M				M
B8340200	HAM	Hammond Creek at SR 1704 nr Mt. Olive	hog farm area	34.5685	-78.5515	Bladen	FRO	18-50	03030005	C	M	M		M				M
B8360000	NC11	Cape Fear River at NC 11 nr East Arcadia	just dns of Lock and Dam #1	34.3969	-78.2675	Bladen	WIRO	18-(59)	03030005	WS-IV Sw	M+2SM	M	EOM	M	M	M		M
B8441000	LVC2	Livingston Creek at Wright Corp. Walkway nr Acme	DWQ ambient stn, dns Wright Corp.	34.3353	-78.2011	Columbus	WIRO	18-64	03030005	C Sw	M	M	EOM		M			M
B8450000	AC	Cape Fear River at Neils Eddy Landing nr Acme	1 mile below IP, DWQ ambient stn	34.3555	-78.1794	Columbus	WIRO	18-(63)	03030005	C Sw	M+2SM	M			M			M
B8465000	DP	Cape Fear River at Intake nr Hooper Hill	At DAK intake, just ups of Black River	34.3358	-78.0534	Brunswick	WIRO	18-(63)	03030005	C Sw	M+2SM	M	EOM		M			M
B8470000	SR	South River at US 13 nr Cooper	dns Dunn runoff	35.1560	-78.6401	Sampson	FRO	18-68-12-(0.5)	03030006	C Sw	M	M		M				M
B8604000	GCO	Great Coharie Creek at SR 1214 nr Butler Crossroads	8 miles dns Clinton WWTP, nonpoint impacts	34.9186	-78.3887	Sampson	FRO	18-68-1	03030006	C Sw	M	M	EOM		M			M
B8610001	LCO	Little Coharie Creek at SR 1207 nr Ingold	Just ups Great Coharie Ck, hog ops in watershed	34.8347	-78.3709	Sampson	FRO	18-68-1-17	03030006	C Sw	M	M		M				M
B8740000	6RC	Six Runs Creek at SR 1003 nr Ingold	Just ups Black River, hog operations in watershed	34.7933	-78.3113	Sampson	FRO	18-68-2-(11.5)	03030006	C Sw ORW+	M	M	EOM	M				M
B8981000	COL	Colly Creek at NC 53 at Colly	Hog operations in watershed	34.4641	-78.2569	Bladen	FRO	18-68-17	03030006	C Sw	M	M	EOM	M				M
B9000000	B210	Black River at NC 210 at Still Bluff	1st bridge ups of Cape Fear River	34.4312	-78.1441	Pender	WIRO	18-68	03030006	C Sw ORW+	M	M			M			M
B9030000	IC	Cape Fear River ups Indian Creek nr Phoenix	Dns DAK, BASF, and Fortron	34.3021	-78.0137	Brunswick	WIRO	18-(63)	03030005	C Sw	M+2SM	M			M			M
B9050025	NAV	Cape Fear River at Navassa dns of RR bridge	dns Progress Energy and Leland Ind. Pk	34.2594	-77.9877	Brunswick	WIRO	18-(71)	03030005	SC	M+2SM	M	EOM	M	M			M
B9050100	HB	Cape Fear River at S. end of Horseshoe Bend nr Wilmington	Ups NE Cape Fear River	34.2437	-77.9698	Brunswick	WIRO	18-(71)	03030005	SC	M+2SM	M			M			M
B9090000	NC403	NE Cape Fear River at NC 403 nr Williams	Dns Mt. Olive WWTP, DWQ ambient stn	35.1784	-77.9807	Duplin	WIRO	18-74-(1)	03030007	C Sw	M	M			M			M
B9130000	PB	Panther Branch (Creek) nr Faison	Sample from Bay Valley access Rd, dns Bay Valley wwtp	35.1345	-78.1363	Duplin	WIRO	18-74-19-3	03030007	C Sw	M	M			M			M
B9191000	GS	Goshen Swamp at NC 11 and NC 903 nr Kornegay	Major trib to NE CFR, Ag. and Hog ops in watershed	35.0281	-77.8516	Duplin	WIRO	18-74-19	03030007	C Sw	M	M		M				M

[1] Field Measurements include: Temperature, Dissolved Oxygen, pH, and Conductivity. M=Monthly, M+2SM=Monthly with twice monthly summer sampling. Summer includes the months of May, June, July, August, and September. Twice monthly samples are to be collected at least ten days apart except when extenuating conditions arise.

[2] Nutrient Sampling includes: Ammonia as N (NH3), Nitrate/Nitrite as N (NO2/NO3), Total Kjeldahl Nitrogen (TKN), and Total Phosphorus as P (TP)

[3] Metals Sampling: EOM=Every Other Month sample collection (February, April, June, August, October, and December) for the following metals: Aluminum (Al), Arsenic (As), Cadmium (Cd), Chromium (Cr), Copper (Cu), Iron (Fe), Lead (Pb), Mercury (Hg), Nickel (Ni), and Zinc (Zn). All analyses will be for total metals. Metals monitoring was suspended per DWQ's April 2010 memorandum at the agreement of DWQ and the LCFRP.

Table A-1 Continued - LCFRP Sampling Stations, Parameters and Sampling Frequency

Station Number	LCFRP Station ID	Location Description	Station Comments	Latitude (dd.dddd)	Longitude (dd.dddd)	County	Region	Index	8 Digit HUC	Stream Class	[1]Field Measurements	[2]Nutrients	[3]Metals	Lab Turbidity	TSS	Chloro-phyll *a*	Entercocci	Fecal Coliform
B9191500	SAR	NE Cape Fear River SR 1700 nr Sarecta	Dns Guilford Mills and Cogentrix WWTPs	34.9801	-77.8622	Duplin	WIRO	18-74-1	03030007	C Sw	M	M	EOM		M			M
B9430000	ROC	Rockfish Creek at US 117 nr Wallace	Ups Wallace WWTP2	34.7168	-77.9795	Duplin	WIRO	18-74-29	03030007	C Sw	M	M			M			M
B9460000	LRC	Little Rockfish Creek at NC 11 nr Wallace	Ups Wallace WWTP1, benthic stn	34.7224	-77.9814	Duplin	WIRO	18-74-29-6	03030007	C Sw	M	M	EOM	M	M			M
B9490000	ANC	Angola Creek at NC 53 nr Maple Hill	benthic stn	34.6562	-77.7351	Pender	WIRO	18-74-33-3	03030007	C Sw	M	M		M				M
B9500000	BCRR	Burgaw Canal (Creek) at SR 1345 Wright St. at Burgaw	Ups Burgaw WWTP	34.5633	-77.9348	Pender	WIRO	18-74-39	03030007	C Sw	M	M		M	M	M		M
B9520000	BC117	Burgaw Creek at US 117 at Burgaw	DWQ ambient stn, dns Burgaw WWTP	34.5637	-77.9220	Pender	WIRO	18-74-39	03030007	C Sw	M	M	EOM	M	M	M		M
B9580000	NCF117	NE Cape Fear River at US 117 at Castle Hayne	DWQ ambient stn, dns Elementis Chromium wwtp	34.3637	-77.8965	New Hanover	WIRO	18-74-(47.5)	03030007	B Sw	M	M	EOM		M			M
B9670000	NCF6	NE Cape Fear River Nr Wrightsboro	Below GNF and Arteva WWTPs	34.3171	-77.9538	New Hanover	WIRO	18-74-(52.5)	03030007	C Sw	M+2SM	M			M			M
B9720000	SC-CH	Smith Creek at US 117 and NC 133 at Wilmington	Dns Smith Ck WWTP, urban runoff	34.2586	-77.9391	New Hanover	WIRO	18-74-63	03030007	C Sw	M							M
B9790000	BRR	Brunswick River dns NC 17 at park nr Belville	Park access from SR 133, dns Belville WWTP	34.2214	-77.9787	Brunswick	WIRO	18-77	03030005	SC	M	M			M		M	
B9795000	M54	Cape Fear River at Channel Marker 54	Dns Wilmington Southside WWTP	34.1393	-77.9460	New Hanover	WIRO	18-(71)	03030005	SC	M+2SM	M	EOM		M		M	
B9800000	M61	Cape Fear River at Channel Marker 61 at Wilmington	Dns Wilmington Northside WWTP, DWQ ambient stn	34.1938	-77.9573	New Hanover	WIRO	18-(71)	03030005	SC	M+2SM	M		M	M	M	M	
B9850100	M35	Cape Fear River at Channel Marker 35	Ups Carolina Beach WWTP	34.0335	-77.9370	Brunswick	WIRO	18-(71)	03030005	SC	M+2SM	M	EOM		M		M	
B9910000	M23	Cape Fear River at Channel Marker 23	Dns Carolina Beach WWTP	33.9456	-77.9696	Brunswick	WIRO	18-(87.5)	03030005	SA HQW	M+2SM	M	EOM		M		M	
B9921000	M18	Cape Fear River at Channel Marker 18	Nr Mouth of Cape Fear River	33.9130	-78.0170	Brunswick	WIRO	18-88-3.5	03030005	SC	M+2SM	M	EOM	M	M	M	M	

[1] Field Measurements include: Temperature, Dissolved Oxygen, pH, and Conductivity. M=Monthly, M+2SM=Monthly with twice monthly summer sampling. Summer includes the months of May, June, July, August, and September. Twice monthly samples are to be collected at least ten days apart except when extenuating conditions arise.

[2] Nutrient Sampling includes: Ammonia as N (NH3), Nitrate/Nitrite as N (NO2/NO3), Total Kjeldahl Nitrogen (TKN), and Total Phosphorus as P (TP)

[3] Metals Sampling: EOM=Every Other Month sample collection (February, April, June, August, October, and December) for the following metals: Aluminum (Al), Arsenic (As), Cadmium (Cd), Chromium (Cr), Copper (Cu), Iron (Fe), Lead (Pb), Mercury (Hg), Nickel (Ni), and Zinc (Zn). All analyses will be for total metals. Metals monitoring was suspended per DWQ's April 2010 memorandum at the agreement of DWQ and the LCFRP.

APPENDIX B - SAMPLE COLLECTION AND ANALYSIS

Sample Collection Procedures

Sample collection shall be performed by trained personnel with NC DWQ certified laboratories in accordance with the DWQ NPDES Discharge Monitoring Coalition Program Field Monitoring Guidance Document (May 2008) and subsequent documents. Alternate collection procedures may be considered if reported to and approved by the DWQ Coalition Coordinator(s) prior to use. Any approved alternate sampling procedures will be documented in writing by the LCFRP.

Laboratory Analysis

All laboratory analyses shall be performed at a DWQ certified laboratory using approved methods as prescribed by 40 CFR, part 136 or other methods certified by the DWQ Laboratory Certification Branch (http://portal.ncdenr.org/web/wq/lab/cert), or the Director of DWQ. Section 40 of the Code of Federal Regulations part 136 (40CFR136) can be accessed on the web at the following address: http://portal.ncdenr.org/web/wq/lab/cert/nonfield/rules.

Reporting levels will be at least as stringent as the target reporting levels used by the DWQ Laboratory. For guidance purposes, Table B-1 lists target reporting levels for each parameter based on the reporting levels of the DWQ Laboratory. The lowest possible analytical limits for all the parameters should be pursued.

Table B-1 DWQ Laboratory Reporting Limits

Parameters	Target Reporting Level	Comments
Temperature		Resolution to 0.1 degree Celsius
Dissolved Oxygen		Report results to the nearest 0.1 mg/l.
pH		Meters should be calibrated to measure a pH range of at least 4.01 to 9.18. Report results to the nearest 0.1 pH units.
Specific Conductivity		Report results to the nearest whole µS/cm at 25 °C.

Table B-1 Continued - DWQ Laboratory Reporting Limits and Methods

Parameters	Target Reporting Level	Comments
Turbidity	1.0 NTU	
TSS	6.2 mg/L	
Entercocci		
Fecal Coliform	1 colony/100 mL	At least 3 dilutions should be used to achieve optimum colony counts per membrane filter of 20-60 colonies.
Chlorophyll *a*	1 μg/L	Report Chlorophyll *a* values free from pheophytin and other chlorophyll pigments. (Not listed in 40 CFR 136) Analysis by HPLC is not approved by DWQ.
Ammonia (NH_3 as N)	0.02 mg/L	Address distillation requirement. See 40CFR136 Table II footnote.
Nitrate+Nitrite as N	0.02 mg/L	
Total Kjeldahl Nitrogen as N	0.20 mg/L	
Total Phosphorus as P	0.02 mg/L	
Al	50 μg/L	
As	2 μg/L	A reporting level of 5 μg/L is acceptable
Cu	2 μg/L	
Cd	1 μg/L	
Cr	10 μg/L	
Fe	50 μg/L	
Pb	10 μg/L	
Hg	0.2 μg/L	
Ni	10 μg/L	
Zn	10 μg/L	

SM=Standard Methods for the Examination of Water and Wastewater, 18^{th}, 19^{th}, and 20th ed.
EPA=EPA Method see 40 CFR 136 (http://www.gpoaccess.gov/cfr/index.html)
APHA=American Public Health Association

Data Qualification Codes

When reporting data, the DWQ's data qualifier codes must be used to provide additional information regarding data quality and interpretation. The current set (codes are subject to change) of qualifier codes to be used is provided in Table B-2. Review the data qualifier codes at least once a year and utilize the most current set being utilized by the DWQ laboratory. Use the following website to check for changes in the qualifier codes: http://portal.ncdenr.org/web/wq/lab/qualityassurance.

Table B-2 Data Qualification Codes For Use With Coalition Data (current as of April 20, 2011)

Data Remark Code	Code Definition
A	Value reported is the mean (average) of two or more determinations. This code is to be used if the results of two or more discrete and separate samples are averaged. These samples shall have been processed and analyzed independently (e.g. field duplicates, different dilutions of the same sample). This code is not required for BOD or coliform reporting since averaging multiple dilutions for these parameters is fundamental to those methods.
B	Results based upon colony counts outside the acceptable range and should be used with caution. This code applies to microbiological tests and specifically to **membrane filter (MF)** colony counts. It is to be used if less than 100% sample was analyzed and the colony count is generated from a plate in which the number of colonies exceeds the ideal ranges indicated by the method. These ideal ranges are defined in the method as: *Fecal coliform or Enterococcus bacteria: 20-60 colonies* *Total coliform bacteria: 20-80 colonies* B1. Countable membranes with less than 20 colonies. Reported value is estimated or is a total of the counts on all filters reported per 100 ml. B2. Counts from all filters were zero. The value reported is based on the number of colonies per 100 ml that would have been reported if there had been one colony on the filter representing the largest filtration volume (reported as a less than "<" value). B3. Countable membranes with more than 60 or 80 colonies. The value reported is calculated using the count from the smallest volume filtered and reported as a greater than ">" value. B4. Filters have counts of both >60 or 80 and <20. Reported value is estimated or is a total of the counts on all filters reported per 100 ml. B5. Too many colonies were present; too numerous to count (TNTC). TNTC is generally defined as >150 colonies. The numeric value represents the maximum number of counts typically accepted on a filter membrane (60 for fecal or enterococcus and 80 for total), multiplied by 100 and then divided by the smallest filtration volume analyzed. This number is reported as a greater than value. B6. Estimated Value. Blank contamination evident. B7. Many non-coliform or non-enterococcus colonies or interfering non-coliform or non-enterococcus growth present. In this competitive situation, the reported value may under-represent actual density. <u>Note</u>: A "B" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., B1, B2, etc.). <u>Note</u>: A "J2" should be used for spiking failures.

Data Remark Code	Code Definition
BB	This code applies to **most probable number (MPN)** microbiological tests. 1. No wells or tubes gave a positive reaction. Value based upon the appropriate MPN Index and reported as a less than "<" value. 2. All wells or tubes gave positive reactions. Value based upon the MPN Index and reported as a greater than ">" value. Note: A "BB" value shall be accompanied by justification for its use denoted by the numbers listed above (e.g., BB1, BB2, etc.).
C	Total residual chlorine was present in sample upon receipt in the laboratory; value is **estimated**. Generally applies to cyanide, phenol, NH_3, TKN, coliform, and organics
G	A single quality control failure occurred during biochemical oxygen demand (BOD) analysis. The sample results should be used with caution. G1. The dissolved oxygen (DO) depletion of the dilution water blank exceeded 0.2 mg/L. G2. The bacterial seed controls did not meet the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L. G3. No sample dilution met the requirement of a DO depletion of at least 2.0 mg/L and/or a DO residual of at least 1.0 mg/L. G4. Evidence of toxicity was present. This is generally characterized by a significant increase in the BOD value as the sample concentration decreases. The reported value is calculated from the highest dilution representing the maximum loading potential and should be considered an **estimated** value. G5. The glucose/glutamic acid standard exceeded the range of 198± 30.5 mg/L. G6. The calculated seed correction exceeded the range of 0.6 to 1.0 mg/L. G7. Less than 1 mg/L DO remained for all dilutions set. The reported value is an **estimated** greater than value and is calculated for the dilution using the least amount of sample. G8. Oxygen usage is less than 2 mg/L for all dilutions set. The reported value is an **estimated** less than value and is calculated for the dilution using the most amount of sample. G9. The DO depletion of the dilution water blank produced a negative value.
J	**Estimated** value; value may not be accurate. This code is to be used in the following instances: J1. Surrogate recovery limits have been exceeded; J2. The reported value failed to meet the established quality control criteria for either precision or accuracy; J3. The sample matrix interfered with the ability to make any accurate determination; J4. The data is questionable because of improper laboratory or field protocols (e.g. composite sample was collected instead of grab, plastic instead of glass container) J5. Temperature limits exceeded (samples frozen or >6° C) during transport or not verifiable (e.g., no temperature blank provided);, non-reportable for NPDES compliance monitoring. J6. The laboratory analysis was from an unpreserved or improperly chemically preserved sample. The data may not be accurate. J7. This qualifier is used to identify analyte concentration exceeding the upper calibration range of the analytical instrument/method. The reported value should be considered estimated. J8. Temperature limits exceeds (samples frozen or >6°C during storage. The data may not be accurate. J9. The reported value is determined by a **one-point estimation** rather than against a regression equation. The estimated concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. J10. Unidentified peak; estimated value. J11. The reported value is determined by a **one-point estimation** rather than against a regression

Data Remark Code	Code Definition
	equation. The estimated concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. *This code is used when an MDL has not been established for the analyte in question.* J12. The calibration verification did not meet the calibration acceptance criterion for **field parameters**. Note: A "J" value shall not be used if another code applies (ex. N, V, M).
M	Sample and duplicate results are "out of control." The sample is non-homogenous (e.g. VOA soil). The reported value is the lower value of duplicate analyses of a sample.
N	Presumptive evidence of presence of material; **estimated** value. This code is to be used if: N1. The component has been tentatively identified based on mass spectral library search; N2. There is an indication that the analyte is present, but quality control requirements for confirmation were not met (i.e., presence of analyte was not confirmed by alternate procedures). N3. This code shall be used if the level is too low to permit accurate quantification, but the **estimated** concentration is less than the laboratory practical quantitation limit and greater than the laboratory method detection limit. *This code is not routinely used for most analyses.* N4. This code shall be used if the level is too low to permit accurate quantification, but the estimated concentration is less than the laboratory practical quantitation limit and greater than the instrument noise level. *This code is used when an MDL has not been established for the analyte in question.* N5. The component has been tentatively identified based on a retention time standard.
P	Elevated practical quantitation limit (PQL)* due to matrix interference and/or sample dilution.
Q	Holding time exceeded. These codes shall be used if the value is derived from a sample that was received, prepared and/or analyzed after the approved holding time restrictions for sample preparation and analysis. The value does not meet NPDES requirements. Q1. Holding time exceeded prior to receipt by lab Q2. Holding time exceeded following receipt by lab
S	Not enough sample provided to prepare and/or analyze a method-required matrix spike (MS) and/or duplicate (MSD).
U	Indicates that the analyte was analyzed for but not detected above the reported practical quantitation limit (PQL)*. The number value reported with the "U" qualifier is equal to the laboratory's PQL*.
V	Indicates the analyte was detected in both the sample and the associated method blank. Note: The value in the blank shall not be subtracted from the associated samples.
X	Sample not analyzed for this constituent. This code is to be used if: X1. Sample not screened for this compound. X2. Sampled, but analysis lost or not performed-field error X3. Sampled, but analysis lost or not performed-lab error
Y	Elevated PQL* due to insufficient sample size
Z	The presence or absence of the analyte cannot be verified. The sample analysis/results are not reported due to: Z1. Inability to analyze the sample. Z2. Questions concerning data reliability.

*PQL The Practical Quantitation Limit (PQL) is defined as the lowest level achievable among laboratories within specified limits during routine laboratory operation. The Practical Quantitation Limit (PQL) is "about three to five times the method detection limit (MDL) and represents a practical and routinely achievable detection level with a relatively good certainty that any reported value is reliable." (APHA, AWWA, WEF. 1992. Standard Methods for the Examination of Water and Wastewater, 18th ed.)

Data Format for Monthly submittals

Table C-1 provides the required data submittal spreadsheet format. Do not use commas, tabs, pipes or other common file delimiters anywhere in the table. The first row should contain the column headings only. Column headings must include appropriate information on measurement units (mg/l, µg/l, cfu/100ml, etc.). The second row must contain the method code. It is very important that the format of the headings and the number and order of columns is consistent among all monthly submissions. The DWQ station number must be provided (e.g. B6140000). An additional column containing the location description is acceptable as long as it is consistently included. Include a comment column for describing pertinent information related to the sampling event or specific samples. Ensure that there are no missing values for station, date, time, and depth. Place all remark codes in a separate column as demonstrated in Table C-1. If there is no result for a particular parameter leave the cell blank. Screen all data for inappropriate or improbable values, such as a pH of 21.2.

Annual Report

The LCFRP is required to submit an annual report by April 30th for each year the Agreement is in effect. The annual report will summarize all data collected in the past calendar year and contain the following elements:

- Monitoring Station List to include station number, station description, county, accurate coordinates (in decimal degrees to 4 decimal places using NAD83), stream classification, and 8 digit hydrologic unit code (HUC).
- List of all certified laboratories that conducted work for the coalition in the past year and laboratory methods used for all parameters. Summarize any laboratory certification issues for individual parameters.
- Submit a CD that includes all monitoring data for the past year with a statistical summary for each station. These data should be combined into a single table containing the year's reviewed and finalized data, which may be placed on the DWQ web site. The annual statistical summary must describe for each parameter at each location:
  - Number of observations (N)
  - Number of observations less than the laboratory reporting level (N<RL)
  - Identify the water quality standard, action level, or other reference level (Ref)
  - Identify the number of observations that do not meet the reference level (N>Ref) or (N<Ref)
  - Maximum observed value and Minimum observed value
  - Annual arithmetic mean (use a geometric mean for fecal coliform data)
- Include a list of active LCFRP members with authorized representative updates, contact names, email addresses and phone numbers. Identify the facility name and permit number. Provide a list of members that are no longer active in the LCFRP.
- Provide a list of changes in members' names, ownerships, and discharge locations.
- Summarize all quality assurance and quality control issues and any field audits conducted.
- Summarize any significant issues, special studies, or projects.
- Describe any required data collection that was missed and provide an explanation.
- Review the monitoring program and suggest potential MOA modifications.
- Provide the Coalition's Website Address.

Table C-1 File Format For Coalition Data Reporting

				Temp (°C)	Temp_rmk	DO (mg/l)	DO_rmk	pH (su)	pH_rmk	Conductivity (uohm/cm)	Conductivity_rmk	Fecal Coliform (#colonies/100ml)	Fecal Coliform_rmk	Suspended Residue (mg/l)	Suspended Residue_rmk	Turbidity (NTU)	Turbidity_rmk	Chlorophyll a (µg/l)	Chlorophyll_rmk	NH3_N (mg/l)	NH3_N_rmk	TKN_N (mg/l)	TKN_N_rmk	NO2_NO3_N (mg/l)	NO2_NO3_N_rmk	TP_P (mg/l)	TP_P_rmk
Station	Date (m/d/yyyy)	Time (hh:mm)	Depth (m)	10	10 rmk	300	300 rmk	400	400 rmk	94	94 rmk	31613	31613 rmk	530	530 rmk	82079	82079 rmk	70953	70953 rmk	610	610 rmk	625	625 rmk	630	630 rmk	665	665 rmk
A1234567	8/19/2002	15:30	0.1	25.2		7.8		6.9		133		110		45		22		23	Q1	0.1		0.2		0.3			
B9876543	8/20/2002	11:50	0.1	27.2		7.1		7.2		125		30		4		5.6		5		0.14		0.6		0.31			
B9876543	8/20/2002	11:50	1	28		6.5		7		122																	
B9876543	8/20/2002	11:50	2	25		6.7		6.9		119																	
B9876543	8/20/2002	11:50	3	17		5.5		6.7		120																	
C1357924	8/21/2002	16:10	0.1	22.1		3.1		6.2		233		15	B1	55		11											
C0246813	9/1/2002	9:30	0.1	19.7		8.3		7		99		6000	B5	410		36				0.26		0.4		0.57			
C0246813	10/1/2002	11:30	0.1	12		8.9		7.3		115		1200	B3	95	A		X3			0.16	J2	0.2		0.09			

The reporting format table continues with metals and comment columns on the next page.

Table C-1 Continued. File Format For Coalition Data Submittals

Cadmium, Cd (µg/L)	Cadmium, Cd_rmk	Chromium, Cr (µg/L)	Chromium, Cr_rmk	Copper, Cu (µg/L)	Copper, Cu_rmk	Nickel, Ni (µg/L)	Nickel, Ni_rmk	Lead, Pb (µg/L)	Lead, Pb_rmk	Zinc, Zn (µg/L)	Zinc, Zn_rmk	Aluminum, Al (µg/L)	Aluminum, Al_rmk	Iron, Fe (µg/L)	Iron, Fe_rmk	Manganese, Mn (µg/L)	Manganese, Mn_rmk	Arsenic, As (µg/L)	Arsenic, As_rmk	Mercury, Hg (µg/L)	Mercury, Hg_rmk	Comments	Enterococci	Enterococci_rmk
1027	1027 rmk	1034	1034 rmk	1042	1042 rmk	1067	1067 rmk	1051	1051 rmk	1092	1092 rmk	1105	1105 rmk	1045	1045 rmk	1055	1055 rmk	1002	1002 rmk	71900	71900 rmk		61211	61211 rmk
130			11	3		27		4.4		610		10				0.21		12		12				
120		10	U	2	U	25	U	2	U	510		10	U	10	U	0.2	U	10	U	10	U			
																						Secchi depth 1.2 meters		
333		10	U	2	U	25	U	2	U	624		10	U	10	U	0.2	U	10	U	10	U	Nutrient Sample Spilled		
120		10	U	2	U	25	U	2	U	510		10	U	10	U	0.2	U	10	U	10	U	2.5" of rain on 8/31/2002		
120		10	U	2	U	25	U	2	U	510		10	U	10	U	0.2	U	10	U	10	U			

APPENDIX A – LCFRP MONITORING PLAN

Table A-1 LCFRP Sampling Stations, Parameters and Sampling Frequency (Revised)

Station Number	Location Description	Station Comments	Latitude (dd.dddd)	Longitude (dd.dddd)	County	Region	Index	8 Digit HUC	Stream Class	[1]Field Measurements	[2]Nutrients	[3]Metals	Lab Turbidity	TSS	Chlorophyll *a*	Enterococci	Fecal Coliform
B8340050	Browns Creek at NC87 nr Elizabethtown	hog farm area	34.6136	-78.5848	Bladen	FRO	18-45	03030005	C	M	M		M				M
B8340200	Hammond Creek at SR 1704 nr Mt. Olive	hog farm area	34.5685	-78.5515	Bladen	FRO	18-50	03030005	C	M	M		M				M
B8360000	Cape Fear River at NC 11 nr East Arcadia	just dns of Lock and Dam #1	34.3969	-78.2675	Bladen	WIRO	18-(59)	03030005	WS-IV Sw	M+2SM	M	EOM	M	M	M		M
B8441000	Livingston Creek at Wright Corp. Walkway nr Acme	DWQ ambient stn, dns Wright Corp.	34.3353	-78.2011	Columbus	WIRO	18-64	03030005	C Sw	M	M	EOM		M			M
B8450000	Cape Fear River at Neils Eddy Landing nr Acme	1 mile below IP, DWQ ambient stn	34.3555	-78.1794	Columbus	WIRO	18-(63)	03030005	C Sw	M+2SM	M			M			M
B8465000	Cape Fear River at Intake nr Hooper Hill	At DAK intake, just ups of Black River	34.3358	-78.0534	Brunswick	WIRO	18-(63)	03030005	C Sw	M+2SM	M	EOM		M			M
B8470000	South River at US 13 nr Cooper	dns Dunn runoff	35.1560	-78.6401	Sampson	FRO	18-68-12-(0.5)	03030006	C Sw	M	M		M				M
B8604000	Great Coharie Creek at SR 1214 nr Butler Crossroads	8 miles dns Clinton WWTP, nonpoint impacts	34.9186	-78.3887	Sampson	FRO	18-68-1	03030006	C Sw	M	M	EOM		M			M
B8610001	Little Coharie Creek at SR 1207 nr Ingold	Just ups Great Coharie Ck, hog ops in watershed	34.8347	-78.3709	Sampson	FRO	18-68-1-17	03030006	C Sw	M	M		M				M
B8740000	Six Runs Creek at SR 1003 nr Ingold	Just ups Black River, hog operations in watershed	34.7933	-78.3113	Sampson	FRO	18-68-2-(11.5)	03030006	C Sw ORW+	M	M	EOM	M				M
B8920000	South River at SR 1007 nr Kerr	Ups of Black River	34.6402	-78.3116	Sampson	FRO	18-68-12-(8.5)	03030006	C Sw ORW+	M	M		M				M
B8981000	Colly Creek at NC 53 at Colly	Hog operations in watershed	34.4641	-78.2569	Bladen	FRO	18-68-17	03030006	C Sw	M	M	EOM	M				M
B9000000	Black River at NC 210 at Still Bluff	1st bridge ups of Cape Fear River	34.4312	-78.1441	Pender	WIRO	18-68	03030006	C Sw ORW+	M	M			M			M
B9030000	Cape Fear River ups Indian Creek nr Phoenix	Dns DAK, BASF, and Fortron	34.3021	-78.0137	Brunswick	WIRO	18-(63)	03030005	C Sw	M+2SM	M			M			M
B9050025	Cape Fear River at Navassa dns of RR bridge	dns Progress Energy and Leland Ind. Pk	34.2594	-77.9877	Brunswick	WIRO	18-(71)	03030005	SC	M+2SM	M	EOM	M	M			M
B9050100	Cape Fear River at S. end of Horseshoe Bend nr Wilmington	Ups NE Cape Fear River	34.2437	-77.9698	Brunswick	WIRO	18-(71)	03030005	SC	M+2SM	M			M			M
B9090000	NE Cape Fear River at NC 403 nr Williams	Dns Mt. Olive WWTP, DWQ ambient stn	35.1784	-77.9807	Duplin	WIRO	18-74-(1)	03030007	C Sw	M	M			M			M
B9130000	Panther Branch (Creek) nr Faison	Sample from Bay Valley access Rd, dns Bay Valley WWTP	35.1345	-78.1363	Duplin	WIRO	18-74-19-3	03030007	C Sw	M	M			M			M
B9191000	Goshen Swamp at NC 11 and NC 903 nr Kornegay	Major trib to NE CFR, Ag. and Hog ops in watershed	35.0281	-77.8516	Duplin	WIRO	18-74-19	03030007	C Sw	M	M		M				M
B9191500	NE Cape Fear River SR 1700 nr Sarecta	Dns Guilford Mills and Cogentrix WWTPs	34.9801	-77.8622	Duplin	WIRO	18-74-1	03030007	C Sw	M	M	EOM		M			M
B9430000	Rockfish Creek at US 117 nr Wallace	Ups Wallace WWTP2	34.7168	-77.9795	Duplin	WIRO	18-74-29	03030007	C Sw	M	M			M			M
B9460000	Little Rockfish Creek at NC 11 nr Wallace	Ups Wallace WWTP1, benthic stn	34.7224	-77.9814	Duplin	WIRO	18-74-29-6	03030007	C Sw	M	M	EOM	M	M			M
B9490000	Angola Creek at NC 53 nr Maple Hill	benthic stn	34.6562	-77.7351	Pender	WIRO	18-74-33-3	03030007	C Sw	M	M		M				M

APPENDIX A – LCFRP MONITORING PLAN

Table A-1 LCFRP Sampling Stations, Parameters and Sampling Frequency (Revised)

Station Number	Location Description	Station Comments	Latitude (dd.dddd)	Longitude (dd.dddd)	County	Region	Index	8 Digit HUC	Stream Class	[1]Field Measurements	[2]Nutrients	[3]Metals	Lab Turbidity	TSS	Chlorophyll *a*	Enterococci	Fecal Coliform
B9580000	NE Cape Fear River at US 117 at Castle Hayne	DWQ ambient stn, dns Elementis Chromium WWTP	34.3637	-77.8965	New Hanover	WIRO	18-74-(47.5)	03030007	B Sw	M	M	EOM		M			M
B9670000	NE Cape Fear River Nr Wrightsboro	Below GNF and Arteva WWTPs	34.3171	-77.9538	New Hanover	WIRO	18-74-(52.5)	03030007	C Sw	M+2SM	M			M			M
B9720000	Smith Creek at US 117 and NC 133 at Wilmington	Dns Smith Ck WWTP, urban runoff	34.2586	-77.9391	New Hanover	WIRO	18-74-63	03030007	C Sw	M	M		M	M			M
B9790000	Brunswick River dns NC 17 at park nr Belville	Park access from SR 133, dns Belville WWTP	34.2214	-77.9787	Brunswick	WIRO	18-77	03030005	SC	M	M			M		M	
B9795000	Cape Fear River at Channel Marker 54	Dns Wilmington Southside WWTP	34.1393	-77.9460	New Hanover	WIRO	18-(71)	03030005	SC	M+2SM	M	EOM		M		M	
B9800000	Cape Fear River at Channel Marker 61 at Wilmington	Dns Wilmington Northside WWTP, DWQ ambient stn	34.1938	-77.9573	New Hanover	WIRO	18-(71)	03030005	SC	M+2SM	M		M	M	M	M	
B9850100	Cape Fear River at Channel Marker 35	Ups Carolina Beach WWTP	34.0335	-77.9370	Brunswick	WIRO	18-(71)	03030005	SC	M+2SM	M	EOM		M		M	
B9910000	Cape Fear River at Channel Marker 23	Dns Carolina Beach WWTP	33.9456	-77.9696	Brunswick	WIRO	18-(87.5)	03030005	SA HQW	M+2SM	M	EOM		M		M	
B9921000	Cape Fear River at Channel Marker 18	Nr Mouth of Cape Fear River	33.9130	-78.0170	Brunswick	WIRO	18-88-3.5	03030005	SC	M+2SM	M	EOM	M	M	M	M	

[1] Field Measurements include: Temperature, Dissolved Oxygen, pH, and Conductivity. M=Monthly, M+2SM=Monthly with twice monthly summer sampling. Summer includes the months of May, June, July, August, and September. Twice monthly samples are to be collected at least ten days apart except when extenuating conditions arise.

[2] Nutrient Sampling includes: Ammonia as N (NH3), Nitrate/Nitrite as N (NO2/NO3), Total Kjeldahl Nitrogen (TKN), and Total Phosphorus as P (TP)

[3] Metals Sampling: EOM=Every Other Month sample collection (February, April, June, August, October, and December) for the following metals: Aluminum (Al), Arsenic (As), Cadmium (Cd), Chromium (Cr), Copper (Cu), Iron (Fe), Lead (Pb), Mercury (Hg), Nickel (Ni), and Zinc (Zn). All analyses will be for total metals. Metals monitoring was suspended per DWQ's April 2010 memorandum at the agreement of DWQ and the LCFRP.

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Appendix III continued.

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Appendix IV continued.

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Fortron Industries, LLC	NC0082295	Gard Gershmel	VP of Operations and Site Director	Gary Long gary.long@ticona.com	(910) 341-3152	Fortron Industries, LLC 4600 Highway 421 N. PO Box 327 Wilmington, NC 28402

Appendix VI - *Curriculum Vitae of LCFRP Scientists*

Michael A. Mallin

Present Position:
Research Professor, Center for Marine Science, University of North Carolina at Wilmington, 5600 Marvin K. Moss Lane., Wilmington, NC. 28409. Telephone (910) 962-2358
Email: mallinm@uncwil.edu   Website:
http://www.uncw.edu/cmsr/aquaticecology/laboratory/

Research Interests: Ecology of phytoplankton and zooplankton; urban and rural pollution sources**;** nutrient and microbial pollution impacts; water quality management issues.

Education:
Ph.D. Estuarine ecology. University of North Carolina at Chapel Hill. 1992.
M.S. Limnology and freshwater ecology. University of Florida. December 1978.
Bachelor of Science. Botany. Ohio University. June 1975.

Professional Experience:
University of North Carolina at Wilmington. Research Professor 2001-present.
Research Associate Professor 2000 – 2001; Research Scientist 1998-1999; Research Associate 1993-1998; Visiting Assistant Professor 1992 –1993
College of Charleston, Adjunct Professor of Environmental Studies, appointed 2003
University of North Carolina Institute of Marine Sciences. (summer 1992; 1994). Visiting Faculty.
Carolina Power and Light Co. New Hill, N.C(1979-1987). Limnologist.

Professional Societies: American Society for Limnology and Oceanography;
Estuarine Research Federation (Member of ERF Governing Board, 2003-2005);
Southeastern Estuarine Research Society (President, 2004-2006);
North American Lake Management Society

Courses Taught: Estuarine Biology; Planktonology; Limnology; Ecology; Biological Oceanography; River Ecology

Awards: Elected Fellow, American Association for the Advancement of Science 2007; UNCW Information Technology Innovation Award 2002; Aldo Leopold Environmental Leadership Fellow 2001 (Ecological Society of America); Large Community Comprehensive Planning Award 2000, *N. H County Tidal Creeks Program* (American Planning Association).

Peer Reviewed Articles (6 of 74 total):
Mallin, M.A., V.L. Johnson and S.H. Ensign. 2009. Comparative impacts of stormwater runoffon water quality of an urban, a suburban, and a rural stream. *Environmental Monitoring and Assessment* (in press).

Appendix VI continued.

Mallin, M.A., L.B. Cahoon, B.R. Toothman, D.C. Parsons, M.R. McIver, M.L. Ortwine and R.N. Harrington. 2007. Impacts of a raw sewage spill on water and sediment quality in an urbanized estuary. *Marine Pollution Bulletin* 54:81-88.

Mallin, M.A., V.L. Johnson, S.H. Ensign and T.A. MacPherson. 2006. Factors contributing to hypoxia in rivers, lakes and streams. *Limnology and Oceanography* 51:690-701.

Mallin, M.A., M.R. McIver, H.A. Wells, D.C. Parsons and V.L. Johnson. 2005. Reversal of eutrophication following sewage treatment upgrades in the New River Estuary, North Carolina. *Estuaries* 28:750-760.

Mallin, M.A., M.R. McIver, S.H. Ensign and L.B. Cahoon. 2004. Photosynthetic and heterotrophic impacts of nutrient loading to blackwater streams. *Ecological Applications* 14:823-838.

Mallin, M.A., K.E. Williams, E.C. Esham and R.P. Lowe. 2000. Effect of human development on bacteriological water quality in coastal watersheds. *Ecological Applications* 10:1047-1056.

Proceedings, Technical Reports and other publications (5 of 93 total):
Mallin, M.A., M.I. Haltom and B. Song. 2009. *Assessing Fecal Bacteria Sources in the Wrightsville Beach, N.C. Area: Final Report*. CMS Report 09-04, Center for Marine Science, University of North Carolina at Wilmington, Wilmington, N.C.

Mallin, M.A., M.R. McIver, M.I.H. Spivey and B. Song. 2009. *Environmental Quality of Wilmington and New Hanover County Watersheds, 2008*. CMS Report 09-03, Center for Marine Science, University of North Carolina at Wilmington, Wilmington, N.C.

Mallin, M.A. and M.R. McIver. 2009. New River Estuary Water Quality 2007, UNCW-CMS Report 09-02. *Report to:* Environmental Management Division, US Marine Corps, Camp Lejeune, N.C. Center for Marine Sciences, University of North Carolina Wilmington, Wilmington, N.C.

Mallin, M.A., M.I.H. Spivey and B. Song. 2009. *Sources of Fecal Bacterial Pollution to Upper Pages Creek, N.C.* Report to Coastal Planning & Engineering of North Carolina, Inc. UNCW-CMS Report 09-01, Center for Marine Science, University of North Carolina at Wilmington, Wilmington, N.C.

Mallin, M.A. 2006. Wading in Waste. *Scientific American* 294:52-59.

Presentations to Professional Organizations: 187 total
Editorial Board Member: Journal of Experimental Marine Biology and Ecology; Coastal and Estuarine Science News (CESN)

Appendix VI continued.

Matthew R. McIver

Present Position
May 1995-present
Research Specialist/Laboratory Manager, Aquatic Ecology Laboratory, Center for Marine Science, University of North Carolina at Wilmington, 5600 Marvin Moss Lane, Wilmington, N.C. 28409. Telephone 910-962-2357 (office), 910-686-0018 (home)
mciverm@uncw.edu

Education
Master of Science, Marine Biology - University of North Carolina Wilmington, Wilmington, NC. May 1997.

Bachelor of Science, Microbiology, minor in Genetics - North Carolina State University, Raleigh, NC. December 1991.

Professional Experience
Lecturer and Educator - Have given over 35 presentations regarding water quality research and science careers

Board Of Directors, Cape Fear River Watch. June 2000-August 2002.

Official Biologist, June 1999-Present, Shallotte Point Volunteer Fire Department Annual Flounder Fishing Tournament, Shallotte Point, North Carolina.

Official Biologist, June 2000, Greater Wilmington King Mackerel Tournament, Wilmington North Carolina

Boat Captain for film crew, August 1998. Captained 28 foot vessel for filming of University of North Carolina at Wilmington Documentary, *Currents of Hope: Reclaiming the Neuse River*.

Lower Cape Fear River Program, Member of Technical, Education, and QA/QC committees, June 1995-Present.

Graduate Research Assistant, July 1994-May 1995, University of North Carolina at Wilmington, Department of Biological Sciences. Water quality analyses including water chemistry, physical parameters using multi-parameter water quality probe, fecal coliform bacteriology and field sampling. Performed nutrient limitation bioassays, phytoplankton identification, data compilation, and supervised an undergraduate assistant for the New Hanover County Tidal Creeks Project and The Cape Fear River Project.

Appendix VI continued

Marine Science Instructor, May 1994-2003 (part-time), Carolina Ocean Studies, Carolina Beach, NC. Lead estuarine and offshore marine education field trips for high school and middle school students.

Official Biologist, September 1993-Present, South Brunswick Islands Chamber of Commerce, Shallote, NC, South Brunswick Islands Annual King Mackerel Fishing Tournament. Inspect fish for compliance with tournament rules.

Laboratory Instructor, August 1992-May 1994, University of North Carolina at Wilmington, Department of Biological Sciences.

Technical Skills

SCUBA Openwater I certified, UNCW NITROX certified, UNCW Boat Captain certified (vessels <25 feet),

Computer Skills

Microsoft Excel, Word, Access, Power Point, US EPA STORET, Paint Shop Pro, Web Page Design, Desktop scanner applications, Netscape Navigator.

Peer Reviewed Articles

Tavares, M.E., M.I.H. Spivey, M.R. McIver and M.A. Mallin. (2008) Testing for optical brighteners and fecal bacteria to detect sewage leaks in tidal creeks. *Journal of the North Carolina Academy of Science* 124:91-97.

Mallin, M.A., L.B. Cahoon, B.R. Toothman, D.C. Parsons, M.R. McIver, M.L. Ortwine and R.N. Harrington. (2007) Impacts of a raw sewage spill on water and sediment quality in an urbanized estuary. *Marine Pollution Bulletin* 54:81-88.

Mallin, M.A., M.R. McIver, H.A. Wells, D.C. Parsons and V.L. Johnson. (2005) Reversal of eutrophication following sewage treatment upgrades in the New River Estuary, North Carolina. *Estuaries* 28:750-760.

Mallin, M.A., M.R. McIver, S.H. Ensign and L.B. Cahoon. (2004) Photosynthetic and heterotrophic impacts of nutrient loading to blackwater streams. Ecological Applications 14:823-838.

Mallin, M.A., L.B. Cahoon, M.R. McIver and S.H. Ensign. (2002) Seeking science based nutrient standards for coastal blackwater stream systems. Report No. 341. Water Resources Research Institute of the University of North Carolina, Raleigh, N.C.

Mallin, M.A., M.H. Posey, M.R. McIver, D.C. Parsons, S.H. Ensign and T.D. Alphin. (2002) Impacts and recovery from multiple hurricanes in a Piedmont-Coastal Plain river system. BioScience 52:999-1010.

Mallin, M.A., S.H. Ensign, M.R. McIver, G.C. Shank and P.K. Fowler. (2001) Demographic, landscape, and meteorological factors controlling the microbial pollution of coastal waters. *Hydrobiologia*.

NCDENR

North Carolina Department of Environment and Natural Resources
Division of Water Resources
Water Quality Programs

Pat McCrory Governor	Thomas A. Reeder Director	John E. Skvarla, III Secretary

MEMORANDUM

DATE: January 1, 2014

TO: Certified Laboratories

FROM: Kent Wiggins, Division of Water Resources Laboratory Section Chief

SUBJECT: 2014 North Carolina Wastewater/Groundwater Laboratory Certification Renewal

The Department of Environment and Natural Resources, in accordance with the provisions of NC GS 143-215-.3 (a) (10), 15 NCAC 2H .0800, is pleased to renew certification for your laboratory to perform specified environmental analyses required by EMC monitoring and reporting regulations 15 NCAC 2B .0500, 2H .0900 and 2L .0100, .0200, .0300, and 2N .0100 through .0800.

Enclosed, for your use, is a certificate describing the requirements and limits of your certification. Please review this certificate and attachment to insure that your laboratory is certified for all parameters required to properly meet your certification needs.

Please contact us at 919-733-3908 if you have questions or need additional information.

Enclosure

DENR DWR WQP Laboratory Section NC Wastewater/Groundwater Laboratory Certification Branch
1623 Mail Service Center, Raleigh, North Carolina 27699-1623
Location: 4405 Reedy Creek Road. Raleigh, North Carolina 27607-6445
Phone: 919-733-3908 \ FAX: 919-733-6241
Internet: www.dwqlab.org

Appendix VII continued.

Attachment I

North Carolina Wastewater/Groundwater Laboratory Certification

Certified Parameters Listing

Lab Name:	Aquatic Ecology Lab - UNCW Center for Marine Science	Certificate Number:	638
Address:	5600 Marvin Moss Lane	Effective Date:	1/1/2014
	Wilmington, NC 28409-	Expiration Date:	12/31/2014

The above named laboratory, having duly met the requirements of 15A NCAC 2H.0800, is hereby certified for the measurement of the parameters listed below.

CERTIFIED PARAMETERS

INORGANIC

CHLOROPHYLL a
  EPA 445.0, Rev. 1.2 (Fluorometric) (Aqueous)
CONDUCTIVITY
  SM 2510 B-1997 (Aqueous)
DISSOLVED OXYGEN
  SM 4500 O G-2001 (Aqueous)
pH
  SM 4500 H+B-2000 (Aqueous)
TEMPERATURE
  SM 2550 B-2000 (Aqueous)

This certification requires maintance of an acceptable quality assurance program, use of approved methodology, and satisfactory performance on evaluation samples. Laboratories are subject to civil penalties and/or decertification for infractions as set forth in 15A NCAC 2H.0807.

Appendix VII continued.

STATE OF NORTH CAROLINA DEPARTMENT OF THE ENVIRONMENT AND NATURAL RESOURCES

DIVISION OF WATER RESOURCES
LABORATORY CERTIFICATION PROGRAM

In accordance with the provisions of N.C.G.S. 143-215.3 (a) (1), 143-215.3 (a)(10) and NCAC 2H.0800:

2014

Aquatic Ecology Lab - UNCW Center for Marine Science

Is hereby certified to perform environmental analysis as listed on Attachment I and report monitoring data to DWR for compliance with NPDES effluent, surface water, groundwater, and pretreatment regulations.

By reference 15A NCAC 2H .0800 is made a part of this certificate.

This certificate does not guarantee validity of data generated, but indicates the methodology, equipment, quality control procedures, records, and proficiency of the laboratory have been examined and found to be acceptable.

This certificate shall be valid until 12/31/2014

Certificate No. 638

J. Kent Wiggins

Appendix VIII - *Analytical Methods and SOPs*

I. Laboraotry Analytical Methods

A. Environmental Chemists, Inc.
6602 Windmill Way
Wilmington, NC 28405
910-392-0223
Lab Certification #94

Analyses and Methods:

Total Phosphorus	SM 4500 P E-1999
Kjeldahl Nitrogen	EPA 351.2, Rev. 2.0-1993
Nitrite+Nitrate	EPA 353.2, Rev. 2.0-1993
Total Sus. Solids	SM 2540 D-1997
Fecal Coliform	SM 9222 D-1997
Enterococcus	Enterolert IDEXX
Lab Turbidity	SM 2130 B-2001

B. Environment 1, Inc.
114 Oakmont Drive
Greenville, NC 27858
252-756-6208
Lab Certification #10

Analyses and Methods:

Ammonia Nitrogen	EPA 350.1,Rev2.0-1993

C. Aquatic Ecology Laboratory
UNCW Center for Marine Sci.
5600 Marvin Moss Lane
Wilmington, NC 28409
910-962-2359
Lab Certification #638

Analyses and Methods:

Chlorophyll *a*	EPA 445.0, Rev. 1.2
Temperature	SM 2550 B-2000
Dissolved Oxygen	SM 4500 O G-2001
Specific Conductivity	SM 2510 B-1997
pH	SM 4500 H B-2000

II. Specific lab Standard Operating Procedures for each method can be obtained by emailing a request to Matthew McIver (mciverm@uncw.edu) in the Aquatic Ecology Laboratory.